INTERLABORATORY STUDY 91-1

RESIN AND FATTY ACIDS AND ADSORBABLE ORGANIC HALIDES IN REAGENT WATER AND EFFLUENTS

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RESIN AND FATTY ACIDS AND ADSORBABLE ORGANIC HALIDES IN REAGENT WATER AND EFFLUENTS

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1 SUMMARY AND CONCLUSIONS

Interlaboratory Study 91-1 was initiated as part of an on-going program of laboratory performance management studies conducted by the Quality Management Office of Laboratory Services Branch, Environment Ontario. It assesses the performance of participating laboratories for the analysis of Resin & Fatty Acids (RFA's) and Adsorbable Organic Halides (AOX) in spiked reagent water and in effluents. Eighteen laboratories (government, academic, industry, and commercial) agreed to participate in this study. Results were received from fifteen participants for RFA's and from twelve participants for AOX. The test groups are MISA Analytical Test Group 26[1] and PP3[2] for RFA's and Group PP4[2] for AOX.

The results for the RFA's indicate that most participants are able to detect these parameters in spiked reagent water. Differences among the participants appear primarily due to a difference in Dehydroabietic Acid, the compound in the scan used as the calibration standard. Results in the matrix samples are more variable. The effluent matrix was biologically active and removed most of the RFA's spiked into the samples. This effect had been noted in previous interlaboratory studies[7,8]. The results for the influent matrix samples showed considerable variability among the participants. This "dirty" matrix suggest that many of the participants did not have a method that was suitable for this type of sample. Further method development work for various matrix samples may be appropriate.

The AOX results show reasonable consistency among the participants for the effluent sample (E1) but considerable variation for the influent sample (I1). As no calibration standard or spiked reagent water sample had been provided for this part of the interlaboratory study, it is not possible to determine how much of the variation among the participants is due to a difference in standards. Some variations in sample preparation steps may have contributed to the variability in the influent sample results.

2 INTRODUCTION

Interlaboratory performance studies are conducted to assess the comparability and accuracy of data among different laboratories. These studies are useful for the identification of biases, precision and accuracy problems. Participation in such studies can serve as a guide for improving individual laboratory performance and maintaining performance standards. The Quality Management Office, Laboratory Services Branch, Environment Ontario has instituted an on-going program of interlaboratory studies to assess and enhance the performance of environmental laboratories providing analytical services.

This study was designed to assess the analytical variability among laboratories for the analysis of Resin and Fatty Acids (RFA's) spiked reagent water and in unspiked natural effluents. This test group was chosen from the MISA (Municipal and Industrial Strategy for Abatement) General Regulation[1]. As well, participants were asked to analyze the unspiked reagent water and two effluent samples for Adsorbable Halides (AOX). Participants were requested to use methods which conformed to the MISA analytical principles and protocols given in the General Regulation[1] or the sector-specific regulation[2].

Eighteen laboratories (government, academic, industry, and commercial) agreed to participate in this study. Results were received from fifteen participants for RFA's and twelve participants for AOX. A list of participants is given in Appendix 2. Each participant was assigned a unique identification code to maintain confidentiality.

A set of nine samples was distributed to each of the eighteen participants. The samples consisted of a reagent water blank, two spiked reagent water samples, an unspiked Pulp & Paper Mill effluent sample, two spiked Pulp & Paper mill effluent samples, one unspiked Pulp & Paper Mill influent, and two spiked Pulp & Paper Mill influent samples. Section 3 describes sample preparation, sample distribution, analytical methodology, and data evaluation procedures. Final results are tabled in Appendix 1 and discussed in Section 4.

3 PROCEDURE

3.1 Preparation of Samples

A stock spiking solution consisting of Resin and Fatty Acids was prepared in methanol and sealed into ampoules. The parameter list is given in Table I. Additional RFA's that are not part of the MISA Test Group were included in the stock solution due to their inclusion in previous interlaboratory studies[6-9].

Samples were prepared in 1000 mL amber glass bottles. To each bottle, 1000 Ml of distilled, deionized water was added by weight (1000 g ±2 g). It was assumed that the density of the water equalled 1.0. The unspiked reagent water samples were labelled D1.

TABLE I

Parameter	MISA Test Group
Abietic Acid	26
Chlorodehydroabietic Acid	26
Dehydroabietic Acid	26
Isopimaric Acid	26
Levopimaric Acid	26
Neoabietic Acid	26
Oleic Acid	26
Pimaric Acid	26
Dichlorodehydroabietic Acid	PP3
9,10-Dichlorostearic Acid	N/A
Palustric Acid	N/A
Sandaracopimaric Acid	N/A

The spiked samples were prepared by individually spiking the sample bottles with the appropriate amount of stock solution using the appropriate size of microlitre syringe. Target values are included in the tables in Appendix 1. The Low Spike was labelled D2 and the High Spike was labelled D3.

Pulp and Paper Mill bulk effluent and influent samples were used for this study. The effluent and influent were separately collected in a stainless steel tank and stirred overnight to form homogenous mixtures using a stirrer with a Teflon shaft and propeller. 1000 mL (1000 g ±2 g) of effluent or influent was poured into amber glass bottles while continuously stirring the bulk sample.

The unspiked effluent was labelled E1, the Low Spike was labelled E2 and the High Spike was labelled E3. Spiking levels for the effluent samples were the same as for the corresponding reagent water samples. The unspiked influent was labelled I1. The spiked influent samples were all spiked at the same level. Samples were labelled either I2 or I3, and participants were provided with one of each. The same spiking procedure was used for the effluent and influent samples as used for the reagent water samples described above. Any possible error introduced by the density of the effluent was considered too small to be significant.

3.2 Sample Distribution

Samples were packed and stored overnight at 4°C. Samples were shipped the following day by Purolator Courier to the participating laboratories. A list of the laboratories receiving sample sets is given in Appendix 2. Samples were shipped on February 20, 1991. A copy of all correspondence is also included in Appendix 2.

One participant telephoned to report receiving one sample minus a cap. A replacement sample was shipped on February 21, 1991. One laboratory contacted the QMO to withdraw from the study due to workload conflicts. Another participant, who was only to participate for AOX analysis, withdrew due to instrument failure.

3.3 Analytical Methodology

Participating laboratories were requested to analyze the samples using routine in-house methods that complied with the principles and protocols outlined in Schedule 3 of the MISA General Regulation[1] or Schedule 2 of the Development Document for the Pulp and Paper Sector[2]. Participants were requested on the report form provided (Appendix 2) to summarize their Sample Preparation Principles, Instrumental Measurement Method Principles, and indicate the Gas Chromatographic Column used for the RFA analysis. All participants were assigned a unique identification code.

3.4 Data Evaluation Techniques

Results were submitted to the Quality Management Office, LSB in written form. All data were manually entered by laboratory code into an electronic spreadsheet.

The participating laboratories who reported results were mailed a copy of the tables of results on June 10, 1991. Several participants reported corrections to their data. Both values are included in the tables, though the corrected result is used in the evaluation.

The mean, median and standard deviation were calculated for each parameter in each sample and are included in Tables 1-3 for RFA's and Table 7 for AOX, Appendix 1. Results of the spiked RFA samples were converted to percent recovery of the target value and are given in Tables 4-6A, Appendix 1. The spiked effluent samples were not corrected for any background levels of RFA's as no significant levels of RFA's were detected in the unspiked sample (E1). Due to the variable background levels of RFA's detected in the unspiked influent sample (I1), the results converted to percent recovery are calculated both "uncorrected" (Table 6) and "corrected" (Table 6A).

The RFA results as converted to percent recovery of the design value are presented as bar graphs in Figures 1-12. The results are arranged in elution order from left to right, based on a DB-1 fused silica GC column. For the spiked influent samples (I2 and I3), the "corrected" results were used.

The results for Dehydroabietic Acid (DHA) in the spiked reagent water samples (D2 and D3) were evaluated using the King-Selliah technique[3,4]. This Automated Evaluation Technique[4] is summarized as follows:

1. Two samples at different concentrations are split among a number of laboratories and analyzed as requested. Results are entered into an electronic spreadsheet and programs (macros) are coded to perform the manipulations. The evaluation technique is performed after the participants have verified their results (see above).

2. High sample data evaluation:
    i) Reject all results which differ from the median (H_m) by more than 10%.
    ii) Calculate median (H), mean and standard deviation (S_h).
    iii) Reinclude results if within 3 times S_h.
    iv) Reiterate ii) and iii) until no further results are included.
    v) Calculate relative standard deviation of final selected results (CV_h).

3. Low sample data evaluation:
    i) Use 3 x CV_h x median(L_m) to exclude possible outliers.
    ii) Calculate median (L), mean and standard deviation (S_l).
    iii) Reinclude results if within 3 times S_l.
    iv) Reiterate ii) and iii) until no further results are included.

4. Paired sample performance criteria:
    i) Examine ratio of S_h/S_l: If <2, use results as reported in concentration units. Otherwise convert results to percent recovery based on target value, if known, or use recovery relative to median value - H, L.
    ii) Prepare paired sample scatter diagrams of all results (Youden plots[5]).
    iii) Calculate perpendicular distances from each point to the two 45° lines (Slope and Intercept error lines) and select the lesser of the two perpendicular distances (PD).
    iv) Determine the median PD.
    v) Determine the mean of all PD values less than 2.5 times the median and use this mean to estimate the repeatability S_w.
    vi) Set warning limits for repeatability: 2 times S_w.
    vii) Set control limits for repeatability: 3 times S_w.
    viii) Set warning limits for possible bias: 3 times S_w (same as vii).

ix) Set control limits for possible bias: 4.5 times S_w.

5. Code performance and summarize in table
    - i) In upper left or lower right quadrant - Erratic
    - ii) In lower left or upper right quadrant - Biased Low or High
    - iii) On horizontal or vertical axis - Out of Control
    - iv) On diagonal line through origin - Slope or Standard problem
    - v) On diagonal line not through origin - Intercept or blank problems.

The performance evaluation of each participant is summarized in Table 8. The graphical results are presented in Figure 13. This technique was applied only to DHA, as this is the parameter in the scan that has a calibration standard. All other parameters, when using an FID detector, are quantitated based on their relative response factors to DHA[10]. Variability among the participants for DHA will affect the variability for the other parameters, and this technique is considered not suitable for the remaining parameters in the scan.

As AOX analysis was only required on unspiked samples, the results could not be converted to percent recovery of a design value. A statistical summary is included in Table 7 for the full data set. The Dixon Test for Outliers[11] was applied to this data set. A statistical summary of the modified data set is also included in Table 7.

4 DISCUSSION

OVERVIEW OF INTERLABORATORY PERFORMANCE - RESIN & FATTY ACIDS

The performance among the participants was reasonably acceptable for the spiked reagent water samples. Recovery for most of the participants was between 75-120% for most parameters in samples D2 and D3. Only one lab (6018) was biased high for most parameters in these samples. Several laboratories had low recoveries for individual parameters, with Laboratory 6020 showing a particularly low bias. When the results for Dehydroabietic Acid (DHA) were evaluated using the King-Selliah Technique[3,4], most participants had acceptable performance for this parameter (Table 8 and Figure 13). Except for Laboratory 6002, the other outlying laboratories demonstrated concentration dependant error, i.e. a difference in their DHA standard. This effect has been noted as the major source of variation among laboratories in previous interlaboratory studies[6-9]. See discussion of individual laboratory performance for possible source of Laboratory 6002's Out of Control performance.

Performance among the participating laboratories was variable for the spiked Pulp & Paper Mill Effluent samples (E2 and E3). Most participants did not detect any of the spiked parameters in the Low Spike (E2). Recovery for the High Spike (E3) generally ranged from 40-80%. Both of these samples were spiked at the same level as the reagent water samples. This effluent, and the influent used for the "I" samples, was from a Pulp & Paper Mill that uses a biological treatment for their effluent. When comparing the amount of RFA's present in the influent (I1) to the lack of RFA's present in the effluent (E1), the treatment process appears to be effective in removing these parameters. This suggests that the biologically active material may still be present in the effluent and therefore acted on the RFA's added to samples E2 and E3. This may be the major contributing factor to the low recovery observed by all of the participants. This effect has been noted in previous interlaboratory studies[8,9].

Considerable variation among the participants was noted for the unspiked influent (I1), so it was not possible to assign a consensus value for the background level of RFA's present in the sample. The two spiked samples (I2 and I3) were duplicates. Many of the participants demonstrated considerable within-laboratory variation when analyzing these two samples. This variation made it difficult to allow for background correction when calculating the percent recovery of the spiked RFA's. The percent recovery has been calculated twice for all of the results for these two samples, one set not background corrected (Table 6) and one set background corrected (Table 6A). This matrix could be considered "dirty" and possibly not typical of most samples provided for analysis, as an untreated effluent would not be discharged into the environment. The variation both within and between laboratories appears to indicate that the current method is not sufficiently rugged for "dirty" matrix samples. Further development work to introduce a clean-up step may improve performance for difficult matrix samples.

OVERVIEW OF INTERLABORATORY PERFORMANCE - ADSORBABLE ORGANIC HALIDES (AOX)

The results among the laboratories showed considerable variation for all three samples. There was a surprising number of positive results for sample D1, the reagent water blank. This suggests that greater care is needed to prevent sample contamination.

The results for the effluent sample (E1) indicate that there is a group of laboratories that show good agreement with each other. Using the Dixon Test for Outliers[11], Laboratories 6005, 6008, 6016, and 6019 were removed as outliers. The mean and median of the remaining laboratories show good agreement (Table 7) with a small standard deviation.

Greater variation was noted for the analysis of the influent sample (I1) when all of the participants' results are included (Table 7). When the Dixon Test is used to remove outliers, Laboratories 6002 and 6016 were excluded. The mean and median of the remaining laboratories show good agreement (Table 7), though the standard deviation is greater for sample I1 than it is for sample E1. Sample pre-treatment may be one factor contributing to the greater variability in the results for this sample. Some participants indicated that they filtered this sample, while others did not. Among the participants who provided details of their method, sample volumes ranged from 25 to 100 mL, another possible source of between-laboratory variability. As a calibration standard had not been included in this study, it is not possible to determine what effect calibration differences had on between-laboratory variability.

INDIVIDUAL INTERLABORATORY PERFORMANCE - RESIN & FATTY ACIDS

Laboratory 6001

This laboratory had consistent performance in the two spiked reagent water samples (D2 and D3). They show a slight pattern of decreasing recovery across the scan (Figures 1 and 2). Only one parameter was detected in sample E2 (Low Spike, Effluent). Most parameters were recovered in sample E3 (High Spike, Effluent). This is typical among the participants, as noted in the interlaboratory overview. Performance was variable between the duplicate influent spiked samples (I2 and I3), though performance across the scan was reasonably consistent, except for DHA in sample I3.

Laboratory 6002

This laboratory noted that analysis of the samples had commenced after the 7 day time limit required in the MISA Regulations[1,2]. This may have contributed to the variable performance observed in their results. Recovery in the spiked reagent water samples (D2 and D3) ranged from 0-153%. They were flagged as Out of Control (Table 8) for DHA. The exceedence of the time limit may have contributed to this. Two parameters were detected in sample E2 (Low Spike, Effluent). Most parameters were recovered in sample E3 (High Spike, Effluent). This is typical among the participants, as noted in the interlaboratory overview. Performance was variable between the duplicate influent spiked samples (I2 and I3). High values reported for the unspiked Influent, I1, results in over-correction for background levels in the spiked samples. This results in "negative recovery" for some parameters (Table 6A). While the exceedence of the time limit may have contributed to this variability, some investigation regarding the ruggedness of their method may also be useful.

Laboratory 6003

Performance in the spiked reagent water samples (D2 and D3) was consistent, though low recovery was observed for Palustric/Levopimaric Acids and Neoabietic Acids. This laboratory was biased low in all spiked Effluent and Influent samples (E2, E3, I2 and I3). A Mass Spectrometer was used as the instrumental detector, which does not conform to the MISA General Regulation[1]. Poor performance when analyzing the matrix samples suggests that the method is not suitable for the matrices used in this interlaboratory study.

Laboratory 6005

This laboratory had reasonably consistent performance in the spiked reagent water samples (D2 and D3), though they were slightly low, with recoveries ranging from 40-80%. Two parameters were detected in sample E2 (Low Spike, Effluent). Most parameters were recovered in sample E3 (High Spike, Effluent). This is typical among the participants, as noted in the interlaboratory overview. They demonstrated good performance in the spiked Influent samples (I2 and I3). Variable performance was noted only for Palustric/Levopimaric and Chlorodehydroabietic Acids.

Laboratory 6006

Laboratory 6006 demonstrated good performance in the spiked reagent water samples (D2 and D3), though they were slightly lower in the High Spike (D3). They were one of the few laboratories to recover most of the spiked parameters in the Effluent Low Spike (E2). The only parameter they did not detect in the Effluent High Spike (E3) was Chlorodehydroabietic Acid. They had consistent performance across the scan for this sample. Consistent performance for the duplicate Influent samples (I2 and I3) was demonstrated. Their estimate of the background levels in the unspiked Influent (I1) allowed for recoveries ranging from 95-130% for most parameters. Only for Palustric/Levopimaric and Neoabietic Acids did they have lower recovery.

Laboratory 6008

This laboratory demonstrated variable performance in the spiked reagent water samples (D2 and D3), with many of the parameters not detected in the Low Spike (D2). Using the King-Selliah Technique[3,4], they were flagged as Biased Low for DHA, with a Concentration (or Slope) dependant error. Good recovery was achieved for Abietic Acid relative to the design value. They did not detect anything in sample E2 (Effluent Low Spike). Variable performance across the scan was observed in sample E3 (Effluent High Spike). Variable performance was observed in the Influent duplicate samples (I2 and I3). Further development work may be necessary for this laboratory to successfully analyze this type of matrix.

Laboratory 6009

Reasonably consistent performance was achieved by this laboratory in the spiked reagent water samples (D2 and D3), though Abietic and Neoabietic Acids were not detected in sample D2. As with other participants, lower recovery was noted for Palustric/Levopimaric and Neoabietic Acids. Only one parameter was detected in sample E2 (Low Spike, Effluent). Most parameters were recovered in sample E3 (High Spike, Effluent), except for 9,10-Dichlorostearic Acid. This is typical among the participants, as noted in the interlaboratory overview. This laboratory had consistent performance for most parameters in the duplicate Influent samples (I2 and I3), except for DHA. Their estimate of the background levels in the unspiked Influent (I1) allowed for good recovery of the design values for most parameters in the Influent spiked samples.

Laboratory 6010

Laboratory 6010 demonstrated good performance for most parameters in the spiked reagent water samples (D2 and D3). They were high for Abietic Acid in both samples. Two parameters were detected in sample E2 (Low Spike, Effluent). Most parameters were recovered in sample E3 (High Spike, Effluent). This is typical among the participants, as noted in the interlaboratory overview. Consistent performance was observed for the duplicate Influent samples (I2 and I3). High recovery was observed for the later eluting parameters (Figures 10 and 12), except for Neoabietic Acid.

Laboratory 6012

Variable performance was observed for the spiked reagent water samples (D2 and D3) for this laboratory. Recoveries ranged from 1-155%. Palustric/Levopimaric Acids were particularly under-recovered. This laboratory was able to detect all of the parameters in sample E2 (Effluent Low spike). Higher recoveries were achieved for most parameters in sample E3 (Effluent High Spike). More consistent performance was observed in the duplicate Influent samples (I2 and I3). Oleic Acid appeared to be more variable than the other parameters in the scan. However, this laboratory had submitted a note with their results, indicating that Oleic Acid was co-eluting with Linolenic Acid. They used an HP-1 column, as opposed to the DB-1 column used by the majority of the participants. Using a different column may improve the separation of these two parameters for this laboratory. Palustric/Levopimaric Acids were not detected in these two samples.

Laboratory 6013

This laboratory demonstrated very variable performance in all the samples. A Mass Spectrometer was used as the instrument detector, which does not conform to the MISA General Regulation[1]. A RTX-30 column, which differs from the DB-1 column used by the majority of participants, was used by this laboratory. The variability in performance suggests that their method is not appropriate for this scan. They were also flagged as Biased Low for DHA in the spiked reagent water samples using the King-Selliah Technique[3,4].

Laboratory 6015

Laboratory 6015 demonstrated good performance for the spiked reagent water samples (D2 and D3). Only Palustric/Levopimaric and Neoabietic Acids had lower recoveries. They detected most of the parameters in sample E2 (Effluent Low Spike) and had good performance for most parameters in sample E3 (Effluent High Spike). Oleic and 9,10-Dichlorostearic Acids had low recoveries in sample E3. Consistent performance for most parameters in the duplicate Influent samples (I2 and I3) was demonstrated. Variable performance was observed for Isopimaric and DHA Acids in these samples.

Laboratory 6016

This laboratory had instrument failure and was unable to perform the analysis for Resin and Fatty Acids.

Laboratory 6017

Good performance was observed for most parameters in the spiked reagent water samples (D2 and D3). Palustric/Levopimaric and Neoabietic Acids had lower recoveries. This laboratory obtained good recovery (70%) for Oleic and Pimaric Acids in sample E2 (Effluent Low Spike), which was different from most participants. They detected all the other parameters in the scan except for Palustric/Levopimaric Acids in sample E2. Their performance was not consistent in sample E3 (Effluent High Spike). Their performance was reasonably consistent for the duplicate Influent samples (I2 and I3). Their high estimate of the background level of Palustric/Levopimaric in the unspiked Influent (I1), results in "negative recovery" of Palustric/Levopimaric when the background level is subtracted (Table 6A).

Laboratory 6018

This laboratory was biased high for most of the parameters in the spiked reagent water samples (D2 and D3). Lower recoveries were observed for Oleic and Abietic Acids. When their DHA results were evaluated using the King-Selliah technique[3], they were flagged as Biased High (Table 8). This laboratory used an ECD detector which may require a different application of standards than using a FID detector[10]. This may have contributed to their high bias. They detected all the parameters in the spiked Effluent samples (E2 and E3). They demonstrated consistent within-laboratory performance for the duplicate Influent samples (I2 and I3), though they were biased high. Only for DHA and Abietic Acids did they have inconsistent performance in these samples.

Laboratory 6019

Laboratory 6019 had better recovery in sample D2 (Low Spike, reagent water) than in sample D3 (High Spike, reagent water). The pattern of recovery across the scan showed the same pattern of good recovery of the early and late eluting parameters, and lower recovery of the mid-scan parameters. They were flagged Biased Low for their DHA results using the King-Selliah Technique[3,4] (Table 8). As the quantitation of all of the parameters is done using their relative response factors to DHA, it is unusual that parameters at the beginning and end of the scan have better recovery when the DHA is low. This should be investigated by the laboratory. This same pattern of recovery is also observed in the spiked Effluent samples (E2 and E3) and less so in the spiked Influent samples (I2 and I3). There was variable within-laboratory performance for Oleic and DHA Acids in the spiked Influent samples.

Laboratory 6020

This laboratory did not detect any of the parameters in samples D2 (Low Spike, reagent water) and E2 (Low Spike, Effluent). They detected all the parameters in sample D3 (High Spike, reagent water), and all but Oleic Acid in sample E3 (high Spike, Effluent). However they were biased low in samples D3 and E3. They were flagged as Low or Erratic for their DHA using the King-Selliah Technique[3,4] (Table 8). The results from these four samples suggest that this laboratory's method is not sufficiently sensitive for low-level samples. They demonstrated better recovery and more consistent performance in the duplicate Influent samples (I2 and I3). However there does appear a pattern of decreasing recovery across the scan in these samples.

Laboratory 6021

This laboratory did not participate in the RFA portion of the interlaboratory study.

INDIVIDUAL INTERLABORATORY PERFORMANCE - AOX

Laboratory 6001

This laboratory had a positive result for sample D1, the unspiked reagent water. They pre-filtered all the samples. This may be the source of possible laboratory contamination. Their result for sample E1 was low compared to the interlaboratory mean and median, while their result for sample I1 was slightly higher than the interlaboratory mean and median. They were not excluded when the Dixon Test for Outliers was applied.

Laboratory 6002

As noted with their results for the RFA's above, this laboratory did not analyze the samples within the time limits required by the MISA protocols[1,2]. While they had good agreement with the interlaboratory mean and median for sample E1, they were biased low for sample I1. The delay in analysis may have contributed to this low bias for sample I1. They were not excluded for sample E1 but were excluded for sample I1 when the Dixon Test for Outliers was applied.

Laboratory 6003

They did not report any results for this part of the interlaboratory study.

Laboratory 6005

This laboratory diluted all of the samples by a factor of 200. This has resulted in very low or non-detectable results. This low bias appears to be due to over-dilution of the samples. They were excluded for sample E1 but not for sample I1 when the Dixon Test for Outliers was applied.

Laboratory 6006

Laboratory 6006 noted that their instrument was equipped with an adsorption module only, and therefore samples E1 and I1 were pre-filtered prior to adsorption, possibly affecting the accuracy of their results. They had good agreement with the interlaboratory mean and median for sample E1 but were biased slightly high for sample I1. They were not excluded when the Dixon Test for Outliers was applied.

Laboratory 6008

Laboratory 6008's results for both E1 and I1 are high compared to the interlaboratory mean and median. As no calibration standard was included in this study, it is not possible to determine their source of high bias. They were excluded for sample E1 but not for sample I1 when the Dixon Test for Outliers was applied.

Laboratory 6009

Laboratory 6009's result for sample E1 was lower than the interlaboratory mean and median. Their result for sample I1 had reasonable agreement with the interlaboratory mean and median. They were not excluded when the Dixon Test for Outliers was applied.

Laboratory 6010

This participant had good agreement with the interlaboratory mean and median for sample E1. Their result for sample I1 was biased low compared to the interlaboratory mean and median. They were not excluded when the Dixon Test for Outliers was applied.

Laboratory 6012

Laboratory 6008's results for both E1 and I1 are low compared to the interlaboratory mean and median. As no calibration standard was included in this study, it is not possible to determine their source of low bias. They were not excluded when the Dixon Test for Outliers was applied.

Laboratory 6013

This laboratory did not participate in the AOX portion of the interlaboratory study.

Laboratory 6015

This laboratory did not participate in the AOX portion of the interlaboratory study.

Laboratory 6016

This laboratory reported a very high result for the unspiked reagent water sample (D1). This suggests possible laboratory contamination. This may have also contributed to their high results for samples E1 and I1, as they are considerably higher than all of the other participants. They were excluded for both samples when the Dixon Test for Outliers was applied.

Laboratory 6017

Laboratory 6017's result for sample E1 was low compared to the interlaboratory mean and median. Their result for sample I1 had reasonable agreement with the interlaboratory mean and median. They were not excluded when the Dixon Test for Outliers was applied.

Laboratory 6018

Laboratory 6008's results for both E1 and I1 are low compared to the interlaboratory mean and median. As no calibration standard was included in this study, it is not possible to determine their source of low bias. They were not excluded when the Dixon Test for Outliers was applied.

Laboratory 6019

This laboratory reported a result for the unspiked reagent water sample (D1). This suggests possible laboratory contamination. Their result for sample E1 was very high compared to all of the other participants for this sample and may have been contaminated. Their result for sample I1 was very low compared to the interlaboratory mean and median. No problems in analysis were noted on the report form. The variability in their results suggests that review of their method may be necessary. They were excluded for sample E1 but not for sample I1 when the Dixon Test for Outliers was applied.

Laboratory 6020

This laboratory did not participate in the AOX portion of the interlaboratory study.

Laboratory 6021

This laboratory had instrument failure and was unable to perform the analysis for AOX.

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6 APPENDIX 1 - TABLES AND GRAPHS

TABLE 1	RFA Results for Reagent Water Samples in µg/L
TABLE 2	RFA Results for Pulp & Paper Effluent Samples in µg/L
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TABLE 1 - RESIN AND FATTY ACIDS
REAGENT WATER SAMPLES; RESULTS IN μg/L

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012	6013	6015	6017	6018	6019	6020
D1	OLEIC	N/A	ND	13.3	ND	ND	4	ND	50	ND	< 2	< 4.8	6 <T	< 4	< 10	21	< MDL
D1	PIMARIC	N/A	ND	< MDL	ND	ND	ND	ND	5 <W	ND	< 2	< 0.28	<W	< 4	< 10	1	< MDL
D1	SANDARACOPIMARIC	N/A	ND			ND	ND	ND	5 <W	ND	< 2		<W				
D1	ISOPIMARIC	N/A	ND	< MDL	ND	ND	ND	ND	5 <W	ND	< 2	< 1.6	<W	< 3	< 10	1	< MDL
D1	PALUSTRIC/LEVOPIMARIC	N/A	ND	< MDL	ND	ND	ND	ND	5 <W	ND	< 2	< 0.16	<W	< 3	< 10	1	< MDL
D1	DEHYDROABIETIC	N/A	ND	< MDL	ND	ND	ND	ND	50	ND	< 2	< 0.43	<W	< 5	< 10	2	< MDL
D1	ABIETIC	N/A	ND	< MDL	ND	ND	ND	ND	5 <W	ND	< 2	< 0.18	<W	< 3	< 10	4	< MDL
D1	NEOABIETIC	N/A	ND	< MDL	ND	ND	ND	ND	5 <W	ND	< 2	< 0.14	<W	< 4	< 10	1	< MDL
D1	9,10-DICHLOROSTEARIC	N/A	ND			ND	ND		28 <T	ND	< 2		<W				
D1	1,2-CHLORODEHYDROABIETIC *	N/A	ND	< MDL	ND	ND	ND	ND	5 <W	ND	2	< 0.22	<W	< 5	< 10	1	< MDL
D1	1,4-CHLORODEHYDROABIETIC *	N/A	ND				ND			ND			<W			2	< MDL
D1	12,14-DICHLORODEHYDROABIETIC	N/A	ND	< MDL	ND	ND	ND	ND	5 <W	ND	< 2	< 0.18	<W	< 4	< 10	1	< MDL
D1	HEPTADECANOIC **		ND				ND										
D1	LINOLEIC **		ND			ND	ND	ND	31 <T	ND	< 2						
D1	LINOLENIC **		ND				ND		28 <T	ND	< 2						
D1	PALMITIC **		23				17			ND	2						
D1	PALMITOLEIC **		ND				5			ND							
D1	STEARIC **		17				5			8							

* The spiking stock contained a mixture of the two isomers of Chlorodehydroabietic Acid. The ratios were not specified by the manufacturer. The target value for Chlorodehydroabietic Acid (Total) is entered in the column adjacent to "1,2-Chlorodehydroabietic Acid". For laboratories reporting only a total value for this compound, this result is entered in the row for 1,2-Chlorodehydroabietic Acid. For those laboratories reporting values for the two isomers, the results are entered separately.

** Additional parameters reported by some of the participants. Not included in spiking stock.

# Revised results provided by the participant after initial review of the Table of Results. Revised result is used in all calculations.

@ Laboratory 6012 reported a combined result for Oleic and Linolenic Acids. The result has been divided evenly between the two compounds.

TABLE 1 - RESIN AND FATTY ACIDS
REAGENT WATER SAMPLES; RESULTS IN µg/L

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012 @	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
D2	OLEIC	53	43	<MDL	66.9	ND	66	ND	85	15	30	79.8	46	43	17	51	<MDL	43	36.2	30.02	0	85	15
D2	PIMARIC	76	56	<MDL	79.5	51	75.5	42	85	61	73	61.4	64	57	141	38	<MDL	61.2	59.0	33.90	0	141	15
D2	SANDARACOPIMARIC	76	59			56	82	382	90	59	67		70					63	65.1	16.15	38	90	8
D2	SANDARACOPIMARIC #							38															
D2	ISOPIMARIC	99	67	123	98.5	62	99.5	25	100	89	153	75	96	111	229	46	<MDL	97.25	91.6	54.04	0	229	15
D2	PALUSTRIC/LEVOPIMARIC	108	42	<MDL	16	31	64	ND	45	64	2	21.2	50	ND	130	30	<MDL	30.5	33.0	35.46	0	130	15
D2	DEHYDROABIETIC	100	88	98	103	75	113.5	45	130	97	115	52.8	114	91	187	30	<MDL	97.5	89.3	45.02	0	187	15
D2	ABIETIC	47	36	48	62.4	41	60.5	47	ND	65	53	36.6	55	40	37	15	<MDL	44	39.8	20.56	0	65	15
D2	NEOABIETIC	62	15	18	19.5	ND	33	ND	ND	19	23	13.6	25	13	92	22	<MDL	18.5	19.5	22.53	0	92	15
D2	9,10-DICHLOROSTEARIC	46	20			ND	49		37	41	44		48					41	34.1	17.94	0	49	7
D2	1,2-CHLORODEHYDROABIETIC *	52	12	22	74.5	32	23.5	ND	25	26	43	54.7	33	48	121	25	<MDL	46.5	42.6	30.69	0	121	15
D2	1,4-CHLORODEHYDROABIETIC *		13				27.5			26			26			20							
D2	12,14-DICHLORODEHYDROABIETIC	53	14	40	63.8	43	67.5	34	45	56	20	96.2	65	43	ND	48	<MDL	44	42.4	26.32	0	96.2	15
D2	HEPTADECANOIC **		N/A				ND																
D2	LINOLEIC **		ND			ND	38	ND	ND	ND													
D2	LINOLENIC **		ND				ND		ND	ND	30												
D2	PALMITIC **		31				28.5			ND	7												
D2	PALMITOLEIC **		ND				11			ND													
D2	STEARIC **		21				10.5				12												

* The spiking stock contained a mixture of the two isomers of Chlorodehydroabietic Acid. The ratios were not specified by the manufacturer. The target value for Chlorodehydroabietic Acid (Total) is entered in the column adjacent to "1,2-Chlorodehydroabietic Acid". For laboratories reporting only a total value for this compound, this result is entered in the row for 1,2-Chlorodehydroabietic Acid. For those laboratories reporting values for the two isomers, the results are entered separately.

** Additional parameters reported by some of the participants. Not included in spiking stock.

# Revised results provided by the participant after initial review of the Table of Results. Revised result is used in all calculations.

@ Laboratory 6012 reported a combined result for Oleic and Linolenic Acids. The result has been divided evenly between the two compounds.

TABLE 1 - RESIN AND FATTY ACIDS
REAGENT WATER SAMPLES; RESULTS IN μg/L

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012 @	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
D3	OLEIC	212	160	169	297	81	164	53	245	92	83.5	216	159	164	35	140	80	159	142.6	73.33	35	297	15
D3	PIMARIC	302	180	49	335	204	158	181	300	387	193	184	222	231	467	140	100	193	222.1	109.50	49	467	15
D3	SANDARACOPIMARIC	330	190			230	181	174	325	335	183		248					210	233.3	65.07	174	335	8
D3	ISOPIMARIC	394	210	400	338	338	190	162	375	332	418	229	311	468	767	165	140	332	322.9	160.39	140	767	15
D3	PALUSTRIC/LEVOPIMARIC	431	140	42	19.7	166	177	124	185	341	2	26.2	189	13	575	36	52	124	139.2	152.85	2	575	15
D3	DEHYDROABIETIC	398	320	610	369	342	409	219	455	447	433	124	420	258	677	120	160	369	357.5	163.97	120	677	15
D3	ABIETIC	189	110	260	116	156	134	209	205	319	152	102	202	180	93	14	46	152	153.2	79.52	14	319	15
D3	NEOABIETIC	246	58	126	31.6	76	98	71	85	119	90	44.4	98	77	468	4	14	77	97.3	108.49	4	468	15
D3	9,10-DICHLOROSTEARIC	182	88			108	162		160	247	161		190					161	159.4	52.22	88	247	7
D3	1,2-CHLORODEHYDROABIETIC *	208	44	101	320	143	66	226	185	116	313	173	99	182	577	84	32	182	210.7	123.14	32	577	15
D3	1,4-CHLORODEHYDROABIETIC *		42				70			140			96			73	69						
D3	DICHLORODEHYDROABIETIC	212	84	221	313	167	119	160	180	263	96	392	225	197	440	182	56	182	206.3	109.11	56	440	15
D3	HEPTADECANOIC **		N/A				ND																
D3	LINOLEIC **		20			ND	12	ND	ND	ND	9												
D3	LINOLENIC **		ND				ND		ND	ND	83.5												
D3	PALMITIC **		64				24			ND	8												
D3	PALMITOLEIC **		18				19			ND													
D3	STEARIC **		46				8				10												

* The spiking stock contained a mixture of the two isomers of Chlorodehydroabietic Acid. The ratios were not specified by the manufacturer. The target value for Chlorodehydroabietic Acid (Total) is entered in the column adjacent to "1,2-Chlorodehydroabietic Acid". For laboratories reporting only a total value for this compound, this result is entered in the row for 1,2-Chlorodehydroabietic Acid. For those laboratories reporting values for the two isomers, the results are entered separately.

** Additional parameters reported by some of the participants. Not included in spiking stock.

# Revised results provided by the participant after initial review of the Table of Results. Revised result is used in all calculations.

@ Laboratory 6012 reported a combined result for Oleic and Linolenic Acids. The result has been divided evenly between the two compounds.

TABLE 2 - RESIN AND FATTY ACIDS
PULP AND PAPER MILL EFFLUENT SAMPLES; RESULTS IN μg/L

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012	6013	6015	6017	6018	6019	6020
E1	OLEIC	N/A	24	<MDL	ND	ND	7.5	ND	20 <T	ND	6	<4.8	4 <T	< 4	38	56	<MDL
E1	PIMARIC	N/A	ND	<MDL	ND	ND	ND	ND	<W	ND	< 2	<0.28	4 <T	< 4	< 10	ND	<MDL
E1	SANDARACOPIMARIC	N/A	ND			ND	ND	ND	<W	ND	< 2		<W				
E1	ISOPIMARIC	N/A	ND	<MDL	ND	ND	ND	ND	<W	ND	< 2	<1.6	<W	< 3	< 10	1	<MDL
E1	PALUSTRIC/LEVOPIMARIC	N/A	ND	<MDL	10.1	ND	ND	ND	<W	ND	< 2	173	<W	< 3	< 10	2	<MDL
E1	DEHYDROABIETIC	N/A	ND	<MDL	TR	ND	ND	ND	<W	ND	< 2	1.99	4 <T	< 5	< 10	3	<MDL
E1	ABIETIC	N/A	ND	<MDL	ND	ND	ND	ND	<W	ND	< 2	<0.18	5 <T	< 3	12	3	<MDL
E1	NEOABIETIC	N/A	ND	<MDL	ND	ND	ND	ND	<W	ND	< 2	<0.14	<W	< 4	12	ND	<MDL
E1	9,10-DICHLOROSTEARIC	N/A	ND			ND	ND		<W	ND	< 2		<W				
E1	1,2-CHLORODEHYDROABIETIC *	N/A	ND	<MDL	ND	ND	ND	ND	<W	ND	< 2	<0.22	<W	< 5	< 10	3	<MDL
E1	1,4-CHLORODEHYDROABIETIC *	N/A	ND				ND	ND		ND			<W			3	<MDL
E1	DICHLORODEHYDROABIETIC	N/A	ND	<MDL	ND	ND	ND	ND	<W	13	< 2	<0.18	<W	< 4	< 10	2	<MDL
E1	HEPTADECANOIC						ND										
E1	LINOLEIC		ND			ND	4	ND	<W	ND	6						
E1	LINOLENIC		ND				ND		<W	ND							
E1	PALMITIC		41				18.5			10	21						
E1	PALMITOLEIC		16				14			ND							
E1	STEARIC		10				5.5				< 2						

* The spiking stock contained a mixture of the two isomers of Chlorodehydroabietic Acid. The ratios were not specified by the manufacturer. The target value for Chlorodehydroabietic Acid (Total) is entered in the column adjacent to "1,2-Chlorodehydroabietic Acid". For laboratories reporting only a total value for this compound, this result is entered in the row for 1,2-Chlorodehydroabietic Acid. For those laboratories reporting values for the two isomers, the results are entered separately.

** Additional parameters reported by some of the participants. Not included in spiking stock.

# Revised results provided by the participant after initial review of the Table of Results. Revised result is used in all calculations.

@ Laboratory 6012 reported a combined result for Oleic and Linolenic Acids. The result has been divided evenly between the two compounds.

TABLE 2 - RESIN AND FATTY ACIDS
PULP AND PAPER MILL EFFLUENT SAMPLES; RESULTS IN μg/L

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012 @	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
E2	OLEIC	53	ND	62	ND	ND	7	ND	17	ND	22	22.5	7	2	48	19	<MDL	2	13.8	18.99	<MDL	62	15
E2	PIMARIC	76	ND	<MDL	3	6	20	ND	<W	ND	9	0.14	<W	6	17	2	<MDL	<W	4.2	6.49	<W	20	15
E2	SANDARACOPIMARIC	76	ND			ND	18	ND	<W	ND	2		8					ND	3.5	6.48	ND	18	8
E2	ISOPIMARIC	99	ND	<MDL	ND	ND	20	ND	<W	ND	15	0.8	22	8	18	2	<MDL	<MDL	5.7	8.50	<MDL	22	15
E2	PALUSTRIC/LEVOPIMARIC	108	ND	<MDL	ND	ND	ND	ND	<W	ND	1	118	<W	1.5	5	ND	<MDL	ND	8.4	30.36	ND	118	15
E2	DEHYDROABIETIC	100	ND	<MDL	TR	ND	7	ND	<W	ND	8	2.25	18	2.5	5	6	<MDL	<MDL	3.3	5.01	<MDL	18	15
E2	ABIETIC	47	ND	<MDL	ND	ND	ND	ND	<W	ND	1	0.09	9	1.5	5	7	<MDL	<MDL	1.6	2.94	<MDL	9	15
E2	NEOABIETIC	62	ND	<MDL	TR	ND	12	ND	<W	ND	1	0.07	<W	2	40	ND	<MDL	<MDL	3.7	10.51	<MDL	40	15
E2	9,10-DICHLOROSTEARIC	46	ND			ND	ND		<W	ND	1		8					ND	1.3	2.98	ND	8	7
E2	1,2-CHLORODEHYDROABIETIC *	52	ND	<MDL	5.5	ND	13	ND	<W	16	< 2	< 0.22	12	24	10	7	<MDL	0.11	6.4	8.12	<MDL	24	15
E2	1,4-CHLORODEHYDROABIETIC *		ND				6	ND		ND			<W			1	<MDL						
E2	DICHLORODEHYDROABIETIC	53	13	10.8	4.4	18	35	ND	<W	22	9	15.5	32	24	5	19	<MDL	10.8	13.8	11.24	<MDL	35	15
E2	HEPTADECANOIC **						ND																
E2	LINOLEIC **		ND			ND	29	ND	<W	ND	12												
E2	LINOLENIC **		ND				4		<W	ND	22												
E2	PALMITIC **		58				49			16	83												
E2	PALMITOLEIC **		64				52			13													
E2	STEARIC **		22				14				12												

* The spiking stock contained a mixture of the two isomers of Chlorodehydroabietic Acid. The ratios were not specified by the manufacturer. The target value for Chlorodehydroabietic Acid (Total) is entered in the column adjacent to "1,2-Chlorodehydroabietic Acid". For laboratories reporting only a total value for this compound, this result is entered in the row for 1,2-Chlorodehydroabietic Acid. For those laboratories reporting values for the two isomers, the results are entered separately.

** Additional parameters reported by some of the participants. Not included in spiking stock.

# Revised results provided by the participant after initial review of the Table of Results. Revised result is used in all calculations.

@ Laboratory 6012 reported a combined result for Oleic and Linolenic Acids. The result has been divided evenly between the two compounds.

TABLE 2 - RESIN AND FATTY ACIDS
PULP AND PAPER MILL EFFLUENT SAMPLES; RESULTS IN μg/L

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012 @	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
E3	OLEIC	212	ND	<MDL	ND	ND	16.5	363	15	ND	4.5	24.2	6	4	37	23	<MDL	4.5	32.9	92.04	<MDL	363	15
E3	PIMARIC	302	210	<MDL	46.6	188	235.5	175	180	233	267	214	225	152	345	131	61	188	177.5	89.56	<MDL	345	16
E3	SANDARACOPIMARIC	330	210			193	243.5	155	180	192	230		228					201.5	203.9	29.50	155	243.5	8
E3	ISOPIMARIC	394	230	270	41.9	209	279.5	140	205	215	428	238	274	242	502	146	90	230	234.0	116.67	41.9	502	15
E3	PALUSTRIC/LEVOPIMARIC	431	120	<MDL	9.2	97	167	72	75	177	1	112	121	1.5	386	194	23	97	104.4	102.53	<MDL	386	15
E3	PALUSTRIC/LEVOPIMARIC #											122											
E3	DEHYDROABIETIC	398	250	360	53.1	247	320.5	140	185	236	282	261	321	172	379	135	70	247	227.4	99.74	53.1	379	15
E3	ABIETIC	189	100	157	16.3	109	163	136	80	154	129	106	137	66	32	83	25	106	99.6	48.44	16.3	163	15
E3	NEOABIETIC	246	60	76	TR	43	106.5	62	55	58	70	12.7	73	38	146	ND	8.3	58	53.9	40.04	0	146	15
E3	9,10-DICHLOROSTEARIC	182	ND			ND	ND		ND	6	1		9					ND	2.3	3.68	ND	9	7
E3	1,2-CHLORODEHYDROABIETIC *	208	62	83	39.8	124	106	271	120	96	145	200	11	63	335	87	22	126	146.0	79.78	38	335	15
E3	1,4-CHLORODEHYDROABIETIC *		52				82.5			64			78	63		69	16						
E3	DICHLORODEHYDROABIETIC	212	110	121	32.3	173	235	239	140	254	87	369	189	163	5	156	38	156	154.1	96.31	5	369	15
E3	HEPTADECANOIC **						ND																
E3	LINOLEIC **		ND			ND	38.5	ND	<W	ND	< 2												
E3	LINOLENIC **		ND				10		<W	ND	4.5												
E3	PALMITIC **		53				70.5			20	46												
E3	PALMITOLEIC **		60				86.5			19													
E3	STEARIC **		17				14				5												

* The spiking stock contained a mixture of the two isomers of Chlorodehydroabietic Acid. The ratios were not specified by the manufacturer. The target value for Chlorodehydroabietic Acid (Total) is entered in the column adjacent to "1,2-Chlorodehydroabietic Acid". For laboratories reporting only a total value for this compound, this result is entered in the row for 1,2-Chlorodehydroabietic Acid. For those laboratories reporting values for the two isomers, the results are entered separately.

** Additional parameters reported by some of the participants. Not included in spiking stock.

# Revised results provided by the participant after initial review of the Table of Results. Revised result is used in all calculations.

@ Laboratory 6012 reported a combined result for Oleic and Linolenic Acids. The result has been divided evenly between the two compounds.

TABLE 3 - RESIN AND FATTY ACIDS
PULP AND PAPER MILL INFLUENT SAMPLES; RESULTS IN µg/L

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012 @	6013	6015	6017	6018	6019	6020
I1	OLEIC	N/A	340	1730	97.9	174	209	200	270	122	212	629	174	158	365	282	
I1	PIMARIC	N/A	150	33	16.5	136	150	96	155	182	159	325	173	158	195	86	< MDL
I1	SANDARACOPIMARIC	N/A	180			158	170	116	190	187	113		205				
I1	ISOPIMARIC	N/A	1300	1030	34.4	1230	1417	576	510	1155	1650	803	1553	1212	477	207	400
I1	PALUSTRIC/LEVOPIMARIC	N/A	220	44	ND	360	320	146	<W	481	< 2	498	530	78	596	884	90
I1	DEHYDROABIETIC	N/A	3300	4800	288	3689	3803	1976	4365	3702	4160	5940	4488	3020	2840	100	1100
I1	ABIETIC	N/A	210	183	29.5	233	235	246	265	393	265	324	219	176	428	311	66
I1	NEOABIETIC	N/A	54	65	ND	33	67	54	31 <T	41	43	79.3	83	16	44	75	18
I1	9,10-DICHLOROSTEARIC	N/A	ND						<W		57		30				
I1	1,2-CHLORODEHYDROABIETIC *	N/A	ND	< MDL	ND	351	ND	ND	<W	ND	42	< 0.22	605 I	< 5	205	14	17
I1	1,4-CHLORODEHYDROABIETIC *	N/A	ND				ND			ND			38			1	< MDL
I1	DICHLORODEHYDROABIETIC	N/A	ND	< MDL	ND	ND	ND	ND	<W	20	391	< 0.18	49	< 4	134	10	< MDL
I1	HEPTADECANOIC **						29										
I1	LINOLEIC **		110			ND	54	ND	90	231	15						
I1	LINOLENIC **		ND				ND		<W	ND	212						
I1	PALMITIC **		1100				507			282	735						
I1	PALMITOLEIC **		140				67			93							
I1	STEARIC **		550				270				708						

* The spiking stock contained a mixture of the two isomers of Chlorodehydroabietic Acid. The ratios were not specified by the manufacturer. The target value for Chlorodehydroabietic Acid (Total) is entered in the column adjacent to "1,2-Chlorodehydroabietic Acid". For laboratories reporting only a total value for this compound, this result is entered in the row for 1,2-Chlorodehydroabietic Acid. For those laboratories reporting values for the two isomers, the results are entered separately.

** Additional parameters reported by some of the participants. Not included in spiking stock.

# Revised results provided by the participant after initial review of the Table of Results. Revised result is used in all calculations.

@ Laboratory 6012 reported a combined result for Oleic and Linolenic Acids. The result has been divided evenly between the two compounds.

TABLE 3 - RESIN AND FATTY ACIDS
PULP AND PAPER MILL INFLUENT SAMPLES; RESULTS IN μg/L

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012 @	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
I2	OLEIC	132	420	440	116	299	366	258	400	283	245.5	827	308	289	493	524	120	308	359.2	175.73	116	827	15
I2	PIMARIC	189	300	30	32.3	276	329	193	330	424	289	625	346	289	486	204	120	289	284.9	159.12	30	625	15
I2	SANDARACOPIMARIC	206	350			321	366.5	208	380	449	268		390					358.3	341.6	75.34	208	449	8
I2	ISOPIMARIC	246	1500	1490	54.3	1522	1735	860	715	1423	1850	1132	1824	1500	1100	355	530	1423	1172.7	557.12	54.3	1850	15
I2	PALUSTRIC/LEVOPIMARIC	270	330	61	ND	385	429	230	260	696	< 2	287	539	47	1290	1148	150	287	390.2	391.48	ND	1290	15
I2	DEHYDROABIETIC	249	3500	5800	333	3982	4218	2175	4470	4182	4210	4400	4589	3165	3230	110	1400	3982	3317.6	1632.65	110	5800	15
I2	ABIETIC	118	310	330	31.1	356	384	347	390	618	383	431	348	289	222	317	110	347	324.4	134.95	31.1	618	15
I2	NEOABIETIC	154	100	162	8.2	100	123.5	67	65	118	99	157	157	84	403	116	27	100	119.1	90.29	8.2	403	15
I2	9,10-DICHLOROSTEARIC	114	71			101	157		90	204	147		120					120	127.1	45.57	71	204	7
I2	1,2-CHLORODEHYDROABIETIC *	130	46	180	22.9	426	57	ND	115	134	142	134	665 I	73	742	87	13	134	214.4	237.26	ND	747	15
I2	1,4-CHLORODEHYDROABIETIC *		43				69.5			78			82			79	28						
I2	DICHLORODEHYDROABIETIC	132	82	159	10.8	140	158	99	115	165	490	266	13	142	240	123	26	140	148.6	119.63	10.8	490	15
I2	HEPTADECANOIC **						30																
I2	LINOLEIC **		110			ND	90.5	ND	120	160	15												
I2	LINOLENIC **		ND				ND		<W	ND	245.5												
I2	PALMITIC **		970				524			446	697												
I2	PALMITOLEIC **		150				109			85													
I2	STEARIC **		390				271.5				666												

* The spiking stock contained a mixture of the two isomers of Chlorodehydroabietic Acid. The ratios were not specified by the manufacturer. The target value for Chlorodehydroabietic Acid (Total) is entered in the column adjacent to "1,2-Chlorodehydroabietic Acid". For laboratories reporting only a total value for this compound, this result is entered in the row for 1,2-Chlorodehydroabietic Acid. For those laboratories reporting values for the two isomers, the results are entered separately.

** Additional parameters reported by some of the participants. Not included in spiking stock.

# Revised results provided by the participant after initial review of the Table of Results. Revised result is used in all calculations.

@ Laboratory 6012 reported a combined result for Oleic and Linolenic Acids. The result has been divided evenly between the two compounds.

TABLE 3 - RESIN AND FATTY ACIDS
PULP AND PAPER MILL INFLUENT SAMPLES; RESULTS IN µg/L

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012 @	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
I3	OLEIC	132	370	460	164	275	348	235	405	282	108.5	982	259	286	472	184	140	282	331.4	211.30	108.5	982	15
I3	PIMARIC	189	215	25	27.3	285	332	178	345	427	296	677	371	286	527	178	115	286	285.6	176.56	25	677	15
I3	SANDARACOPIMARIC	206	250			328	367	191	355	445	183		399					341.5	314.8	96.70	183	445	8
I3	ISOPIMARIC	246	1350	1360	45.4	1458	1759	611	695	1405	1940	1193	2016	1390	1100	324	525	1350	1144.8	588.04	45.4	2016	15
I3	PALUSTRIC/LEVOPIMARIC	270	270	<MDL	ND	492	458	231	50	656	< 2	415	668	56	1350	1056	145	270	389.9	406.31	ND	1350	15
I3	DEHYDROABIETIC	249	3200	5300	267	3939	4203	1816	4355	4121	4290	5400	4874	3158	3660	93	1300	3939	3331.7	1705.23	93	5400	15
I3	ABIETIC	118	245	240	24.5	363	353	320	400	602	380	461	324	287	< 10	327	102	324	295.6	158.26	5	602	15
I3	NEOABIETIC	154	75.5	153	ND	108	131	66	60	107	95	180	161	57	443	126	26	107	119.2	102.64	ND	443	15
I3	9,10-DICHLOROSTEARIC	114	70			76	155		80	205	159		120					120	123.6	51.51	70	205	7
I3	1,2-CHLORODEHYDROABIETIC *	130	44	163	17.8	354	65	ND	120	134	151	121	650 I	196	809	73	7.9	151	219.8	241.74	ND	809	15
I3	1,4-CHLORODEHYDROABIETIC *		41				72			83			91			78	27						
I3	DICHLORODEHYDROABIETIC	132	72	156	4.5	111	159	320	110	152	527	272	155	111	257	116	25	116	154.5	125.52	4.5	527	15
I3	DICHLORODEHYDROABIETIC #							90															
I3	HEPTADECANOIC **						29																
I3	LINOLEIC **		100			ND	53	ND	110	132	29												
I3	LINOLENIC **		ND				ND		<W	ND	108.5												
I3	PALMITIC **		940				490			467	715												
I3	PALMITOLEIC **		140				104			83													
I3	STEARIC **		465				244				655												

* The spiking stock contained a mixture of the two isomers of Chlorodehydroabietic Acid. The ratios were not specified by the manufacturer. The target value for Chlorodehydroabietic Acid (Total) is entered in the column adjacent to "1,2-Chlorodehydroabietic Acid". For laboratories reporting only a total value for this compound, this result is entered in the row for 1,2-Chlorodehydroabietic Acid. For those laboratories reporting values for the two isomers, the results are entered separately.

** Additional parameters reported by some of the participants. Not included in spiking stock.

# Revised results provided by the participant after initial review of the Table of Results. Revised result is used in all calculations.

@ Laboratory 6012 reported a combined result for Oleic and Linolenic Acids. The result has been divided evenly between the two compounds.

TABLE 4 - RESIN AND FATTY ACIDS
REAGENT WATER SAMPLES; RESULTS CONVERTED TO PERCENT RECOVERY OF DESIGN

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
D2	OLEIC	53	81%	0%	126%	0%	125%	0%	160%	28%	57%	151%	87%	81%	32%	96%	0%	81%	68%	56.6%	0%	160%	15
D2	PIMARIC	76	74%	0%	105%	67%	99%	55%	112%	80%	96%	81%	84%	75%	186%	50%	0%	81%	78%	44.6%	0%	186%	15
D2	SANDARACOPIMARIC	76	78%			74%	108%	50%	118%	78%	88%		92%					83%	86%	21.2%	50%	118%	8
D2	ISOPIMARIC	99	68%	124%	99%	63%	101%	25%	101%	90%	155%	76%	97%	112%	231%	46%	0%	98%	93%	54.6%	0%	231%	15
D2	PALUSTRIC/LEVOPIMARIC	108	39%	0%	15%	29%	59%	0%	42%	59%	2%	20%	46%	0%	120%	28%	0%	28%	31%	32.8%	0%	120%	15
D2	DEHYDROABIETIC	100	88%	98%	103%	75%	114%	45%	130%	97%	115%	53%	114%	91%	187%	30%	0%	98%	89%	45.0%	0%	187%	15
D2	ABIETIC	47	77%	102%	133%	87%	129%	100%	0%	138%	113%	78%	117%	85%	79%	32%	0%	94%	85%	43.7%	0%	138%	15
D2	NEOABIETIC	62	24%	29%	31%	0%	53%	0%	0%	31%	37%	22%	40%	21%	148%	35%	0%	30%	32%	36.3%	0%	148%	15
D2	9,10-DICHLOROSTEARIC	46	43%			0%	107%		80%	89%	96%		104%					89%	74%	39.0%	0%	107%	7
D2	CHLORODEHYDROABIETIC	52	48%	42%	143%	62%	98%	0%	48%	100%	83%	105%	113%	92%	233%	87%	0%	83%	80%	49.7%	0%	233%	15
D2	DICHLORODEHYDROABIETIC	53	26%	75%	120%	81%	127%	64%	85%	106%	38%	182%	123%	81%	0%	91%	0%	83%	80%	49.7%	0%	182%	15
D3	OLEIC	212	75%	80%	140%	38%	77%	25%	116%	43%	39%	102%	75%	77%	17%	66%	38%	75%	67%	34.6%	17%	140%	15
D3	PIMARIC	302	60%	16%	111%	68%	52%	60%	99%	128%	64%	61%	74%	76%	155%	46%	33%	64%	74%	36.3%	16%	155%	15
D3	SANDARACOPIMARIC	330	58%			70%	55%	53%	98%	102%	55%		75%					64%	71%	19.7%	53%	102%	8
D3	ISOPIMARIC	394	53%	102%	86%	86%	48%	41%	95%	84%	106%	58%	79%	119%	195%	42%	36%	84%	82%	40.7%	36%	195%	15
D3	PALUSTRIC/LEVOPIMARIC	431	32%	10%	5%	39%	41%	29%	43%	79%	0.5%	6%	44%	3%	133%	8%	12%	29%	32%	35.5%	0.5%	133%	15
D3	DEHYDROABIETIC	398	80%	153%	93%	86%	103%	55%	114%	112%	109%	31%	106%	65%	170%	30%	40%	93%	90%	41.2%	30%	170%	15
D3	ABIETIC	189	58%	138%	61%	83%	71%	111%	108%	169%	80%	54%	107%	95%	49%	7%	24%	80%	81%	42.1%	7%	169%	15
D3	NEOABIETIC	246	24%	51%	13%	31%	40%	29%	35%	48%	37%	18%	40%	31%	190%	2%	6%	31%	40%	44.1%	2%	190%	15
D3	9,10-DICHLOROSTEARIC	182	48%			59%	89%		88%	136%	88%		104%					88%	88%	28.7%	48%	136%	7
D3	CHLORODEHYDROABIETIC	208	46%	49%	154%	69%	65%	109%	89%	123%	150%	83%	94%	88%	277%	75%	49%	88%	101%	59.2%	21%	277%	15
D3	DICHLORODEHYDROABIETIC	212	40%	104%	148%	79%	56%	75%	85%	124%	45%	185%	106%	93%	208%	86%	26%	86%	97%	51.5%	26%	208%	15

TABLE 5 - RESIN AND FATTY ACIDS
PULP & PAPER EFFLUENT SAMPLES; RESULTS CONVERTED TO PERCENT RECOVERY OF DESIGN

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
E2	OLEIC	53	0%	117%	0%	0%	13%	0%	32%	0%	42%	42%	13%	4%	91%	36%	0%	4%	26%	35.8%	0%	117%	15
E2	PIMARIC	76	0%	0%	4%	8%	26%	0%	0%	0%	12%	0%	0%	8%	22%	3%	0%	0%	6%	8.5%	0%	26%	15
E2	SANDARACOPIMARIC	76	0%		0%	0%	24%	0%	0%	0%	3%		11%					0%	5%	8.5%	0%	24%	8
E2	ISOPIMARIC	99	0%	0%	0%	0%	20%	0%	0%	0%	15%	1%	22%	8%	18%	2%	0%	0%	6%	8.6%	0%	22%	15
E2	PALUSTRIC/LEVOPIMARIC	108	0%	0%	0%	0%	0%	0%	0%	0%	1%	109%	0%	1%	5%	0%	0%	0%	8%	28.1%	0%	109%	15
E2	DEHYDROABIETIC	100	0%	0%	0%	0%	7%	0%	0%	0%	8%	2%	18%	3%	5%	6%	0%	0%	3%	5.0%	0%	18%	15
E2	ABIETIC	47	0%	0%	0%	0%	0%	0%	0%	0%	2%	0%	19%	3%	11%	15%	0%	0%	3%	6.3%	0%	19%	15
E2	NEOABIETIC	62	0%	0%	0%	0%	19%	0%	0%	0%	2%	0%	0%	3%	65%	0%	0%	0%	6%	17.0%	0%	65%	15
E2	9,10-DICHLOROSTEARIC	46	0%			0%	0%		0%	0%	2%		17%					0%	3%	6.5%	0%	17%	7
E2	CHLORODEHYDROABIETIC	52	0%	0%	11%	0%	37%	0%	0%	31%	2%	0%	23%	46%	19%	15%	0%	0%	12%	15.6%	0%	46%	15
E2	DICHLORODEHYDROABIETIC	53	25%	20%	8%	34%	66%	0%	0%	42%	17%	29%	60%	45%	9%	36%	0%	20%	26%	21.2%	0%	66%	15
E3	OLEIC	212	0%	0%	0%	0%	8%	171%	7%	0%	2%	11%	3%	2%	17%	11%	0%	2.1%	16%	43.4%	0%	171%	15
E3	PIMARIC	302	70%	0%	15%	62%	78%	58%	60%	77%	88%	71%	75%	50%	114%	43%	20%	62.3%	59%	29.7%	0%	114%	15
E3	SANDARACOPIMARIC	330	64%			58%	74%	47%	55%	58%	70%		69%					61.1%	62%	8.9%	47%	74%	8
E3	ISOPIMARIC	394	58%	69%	11%	53%	71%	36%	52%	55%	109%	60%	70%	61%	127%	37%	23%	58.4%	59%	29.6%	11%	127%	15
E3	PALUSTRIC/LEVOPIMARIC	431	28%	0%	2%	23%	39%	17%	17%	41%	0.2%	28%	28%	0.3%	90%	45%	5%	22.5%	24%	23.8%	0.0%	90%	15
E3	DEHYDROABIETIC	398	63%	90%	13%	62%	81%	35%	46%	59%	71%	66%	81%	43%	95%	34%	18%	62.1%	57%	25.1%	13%	95%	1
E3	ABIETIC	189	53%	83%	9%	58%	86%	72%	42%	81%	68%	56%	72%	35%	17%	44%	13%	56.1%	53%	25.6%	9%	86%	15
E3	NEOABIETIC	246	24%	31%	0%	17%	43%	25%	22%	24%	28%	5%	30%	15%	59%	0%	3%	23.6%	22%	16.3%	0%	59%	15
E3	9,10-DICHLOROSTEARIC	182	0%			0%	0%		0%	3%	1%		5%					0.0%	1%	2.0%	0%	5%	7
E3	CHLORODEHYDROABIETIC	208	55%	40%	19%	60%	91%	130%	58%	77%	70%	96%	43%	61%	161%	75%	18%	60.6%	70%	38.4%	18%	161%	15
E3	DICHLORODEHYDROABIETIC	212	52%	57%	15%	82%	111%	113%	66%	120 %	41%	174%	89%	77%	2%	74%	18%	73.6%	73%	45.4%	2%	174%	15

TABLE 6 - RESIN AND FATTY ACIDS
PULP & PAPER INFLUENT SAMPLES; RESULTS CONVERTED TO PERCENT RECOVERY OF DESIGN
RESULTS NOT CORRECTED FOR BACKGROUND LEVELS

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	6005	6006	6008	6009	6010	6012	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
I2	OLEIC	132	318%	333%	88%	227%	277%	195%	303%	214%	186%	627%	233%	219%	373%	397%	91%	233%	272%	133.1%	88%	627%	15
I2	PIMARIC	189	159%	16%	17%	146%	174%	102%	175%	224%	153%	331%	183%	153%	257%	108%	63%	153%	151%	84.2%	16%	331%	15
I2	SANDARACOPIMARIC	206	170%		0%	156%	178%	101%	184%	218%	130%		189%					174%	166%	36.6%	0%	218%	8
I2	ISOPIMARIC	246	610%	606%	22%	619%	705%	350%	291%	578%	752%	460%	741%	610%	447%	144%	215%	578%	477%	226.5%	22%	752%	15
I2	PALUSTRIC/LEVOPIMARIC	270	122%	23%	0%	143%	159%	85%	96%	258%	0%	106%	200%	17%	478%	425%	56%	106%	145%	145.0%	0%	478%	15
I2	DEHYDROABIETIC	249	1406%	2329%	134%	1599%	1694%	873%	1795%	1680%	1691%	1767%	1843%	1271%	1297%	44%	562%	1599%	1332%	655.7%	44%	2329%	15
I2	ABIETIC	118	263%	280%	26%	302%	325%	294%	331%	524%	325%	365%	295%	245%	188%	269%	93%	294%	275%	114.4%	26%	524%	15
I2	NEOABIETIC	154	65%	105%	5%	65%	80%	44%	42%	77%	64%	102%	102%	55%	262%	75%	18%	65%	77%	58.6%	5%	262%	15
I2	9,10-DICHLOROSTEARIC	114	62%			89%	138%		79%	179%	129%		105%					105%	112%	40.0%	62%	179%	7
I2	CHLORODEHYDROABIETIC	130	68%	138%	18%	328%	97%	0%	88%	163%	109%	103%	575%	56%	571%	128%	32%	103%	165%	182.5%	0%	575%	15
I2	DICHLORODEHYDROABIETIC	132	62%	120%	8%	106%	120%	75%	87%	125%	371%	202%	10%	108%	182%	93%	20%	106%	113%	90.6%	8%	371%	15
I3	OLEIC	132	280%	348%	124%	208%	264%	178%	307%	214%	82%	744%	196%	217%	358%	139%	106%	213.6%	251%	160.1%	82%	744%	15
I3	PIMARIC	189	114%	13%	14%	151%	176%	94%	183%	226%	157%	358%	196%	151%	279%	94%	61%	151.3%	151%	93.4%	13%	358%	15
I3	SANDARACOPIMARIC	206	121%			159%	178%	93%	172%	216%	89%		194%					165.8%	153%	46.9%	89%	216%	8
I3	ISOPIMARIC	246	549%	553%	18%	593%	715%	248%	283%	571%	789%	485%	820%	565%	447%	132%	213%	548.8%	465%	239.0%	18%	820%	15
I3	PALUSTRIC/LEVOPIMARIC	270	100%	0%	0%	182%	170%	86%	19%	243%	0.4%	154%	247%	21%	500%	391%	54%	100.0%	144%	150.5%	0%	500%	15
I3	DEHYDROABIETIC	249	1285%	2129%	107%	1582%	1688%	729%	1749%	1655%	1723%	2169%	1957%	1268%	1470%	37%	522%	1581.9%	1338%	684.8%	37%	2169%	15
I3	ABIETIC	118	208%	203%	21%	308%	299%	271%	339%	510%	322%	391%	275%	243%	4%	277%	86%	274.6%	250%	134.1%	4%	510%	15
I3	NEOABIETIC	154	49%	99%	0%	70%	85%	43%	39%	69%	62%	117%	105%	37%	288%	82%	17%	69.5%	77%	66.6%	0%	288%	15
I3	9,10-DICHLOROSTEARIC	114	61%			67%	136%		70%	180%	139%		105%					105.3%	108%	45.2%	61%	180%	7
I3	CHLORODEHYDROABIETIC	130	65%	125%	14%	272%	105%	0%	92%	167%	116%	93%	570%	151%	622%	116%	27%	116.2%	169%	186.0%	0%	622%	15
I3	DICHLORODEHYDROABIETIC	132	55%	118%	3%	84%	120%	68%	83%	115%	399%	206%	117%	84%	195%	88%	19%	87.9%	117%	95.1%	3%	399%	15

TABLE 6A - RESIN AND FATTY ACIDS
PULP & PAPER INFLUENT SAMPLES; RESULTS CONVERTED TO PERCENT RECOVERY OF DESIGN RESULTS CORRECTED FOR BACKGROUND LEVELS

SAMPLE	PARAMETER	SPIKE	6001	6002	6003	8005	6006	6008	6009	6010	6012	6013	6015	6017	6018	6019	6020	MEDIAN	MEAN	STD DEV	MIN	MAX	n
I2	OLEIC	53	61%	-977%	14%	95%	119%	44%	98%	122%	25%	150%	102%	99%	97%	183%	91%	97%	93%	45.9%	-977%	183%	14
I2	PIMARIC	76	79%	-2%	8%	74%	95%	51%	93%	128%	69%	159%	92%	69%	154%	62%	63%	74%	80%	44.7%	-2%	159%	15
I2	SANDARACOPIMARIC	76	83%			79%	95%	45%	92%	127%	75%		90%					86%	86%	23.1%	45%	127%	8
I2	ISOPIMARIC	99	81%	187%	8%	119%	129%	115%	83%	109%	81%	134%	110%	117%	253%	60%	53%	110%	109%	57.3%	8%	253%	15
I2	PALUSTRIC/LEVOPIMARIC	108	41%	6%	0%	9%	40%	31%	96%	80%	0%	-78%	3%	-11%	257%	98%	22%	22%	48%	70.1%	-78%	257%	14
I2	DEHYDROABIETIC	100	80%	402%	18%	118%	167%	80%	42%	193%	20%	-618%	41%	58%	157%	4%	120%	80%	107%	103.4%	-618%	402%	14
I2	ABIETIC	47	85%	125%	1%	104%	126%	86%	106%	191%	100%	91%	109%	96%	-175%	5%	37%	96%	90%	49.2%	-175%	191%	14
I2	NEOABIETIC	62	30%	63%	5%	44%	37%	8%	22%	50%	36%	50%	48%	44%	233%	27%	6%	37%	47%	54.4%	5%	233%	15
I2	9,10-DICHLOROSTEARIC	46	52%			89%	138%	0%	79%	179%	79%		79%					79%	101%	41.9%	0%	179%	7
I2	CHLORODEHYDROABIETIC	52	68%	138%	18%	58%	97%	0%	88%	163%	77%	103%	80%	54%	413%	116%	18%	80%	100%	97.5%	0%	413%	15
I2	DICHLORODEHYDROABIETIC	53	62%	120%	8%	106%	120%	75%	87%	110%	75%	201%	-27%	106%	80%	86%	20%	86%	82%	54.1%	-27%	201%	15
I3	OLEIC	212	23%	-962%	50%	77%	105%	27%	102%	121%	-78%	267%	64%	97%	81%	-74%	106%	77%	93%	63.3%	-962%	267%	12
I3	PIMARIC	302	34%	-4%	6%	79%	96%	43%	101%	130%	72%	186%	105%	68%	176%	49%	61%	72%	80%	54.6%	-4%	186%	15
I3	SANDARACOPIMARIC	330	34%			83%	96%	36%	80%	125%	34%		94%					81%	73%	34.3%	34%	125%	8
I3	ISOPIMARIC	394	20%	134%	4%	93%	139%	14%	75%	102%	118%	159%	188%	72%	253%	48%	51%	93%	98%	69.2%	4%	253%	15
I3	PALUSTRIC/LEVOPIMARIC	431	19%	-16%	0%	49%	51%	31%	19%	65%	0%	-31%	51%	-8%	279%	64%	20%	20%	40%	72.6%	-30.7%	279%	15
I3	DEHYDROABIETIC	398	-40%	201%	-8%	100%	161%	-64%	-4%	168%	52%	-217%	155%	55%	329%	-3%	80%	55%	96%	104.6%	-217%	329%	13
I3	ABIETIC	189	30%	48%	-4%	110%	100%	63%	114%	177%	97%	116%	89%	94%	-358%	14%	31%	89%	77%	49.2%	-358%	177%	14
I3	NEOABIETIC	246	14%	57%	0%	49%	42%	8%	19%	43%	34%	65%	51%	27%	259%	33%	5%	34%	47%	61.9%	0%	259%	15
I3	9,10-DICHLOROSTEARIC	182	61%			67%	136%	0%	70%	180%	89%		79%					75%	97%	44.1%	0%	180%	7
I3	CHLORODEHYDROABIETIC	208	65%	125%	14%	2%	105%	0%	92%	167%	84%	93%	75%	149%	465%	105%	14%	92%	104%	112.4%	0%	465%	15
I3	DICHLORODEHYDROABIETIC	212	55%	118%	3%	84%	120%	68%	83%	100%	103%	206%	80%	83%	93%	80%	19%	83%	86%	46.2%	3%	206%	15

TABLE 7 - ADSORBABLE ORGANIC HALIDES RESULTS IN µg/L

LAB CODE	D1	E1	I1	INSTRUMENT
6001	8.3	52	105	DOHRMANN
6002	<50	74	<60	MITSUBISHI
6003	NOT REPORTED			
6005	0.5	0.1	0	DOHRMANN
6006	ND	72	116	DOHRMANN
6008	<10	120	120	DOHRMANN
6009	4.4	60	76	MITSUBISHI
6010	<10	70	55	DOHRMANN
6012	<10	55	68	MITSUBISHI
6013	N/A	N/A	N/A	
6015	N/A	N/A	N/A	
6016	125	200	535	DOHRMANN
6017	<6	57	79	MITSUBISHI
6018	<10	52	71	MITSUBISHI
6019	23.8	231.3	43.3	MITSUBISHI
6021	NO RESULTS; INSTRUMENT FAILURE			
MEAN	-	76.7	90.6	ALL
MEDIAN	-	65	73.5	
STD DEV	-	66.1	140.7	
n	-	12	12	
MEAN	-	61.5	73.33	OUTLIERS REMOVED (DIXON'S TEST FOR OUTLIERS)
MEDIAN	-	58.5	73.5	
STD DEV	-	9.134	36.040	
n	-	8	10	

TABLE 8 - EVALUATION OF DEHYDROABIETIC ACID
SUMMARY OF PERFORMANCE EVALUATION

LAB CODE	FLAG	DESCRIPTION
6001	A	ACCEPTABLE PERFORMANCE
6002	OC	ONE RESULT ERRATIC
6003	A	ACCEPTABLE PERFORMANCE
6005	A	ACCEPTABLE PERFORMANCE
6006	A	ACCEPTABLE PERFORMANCE
6008	LS	BIASED LOW, PROBABLE SLOPE PROBLEM
6009	WHI	WARNING:BIASED HIGH, PROBABLE INTERCEPT PROBLEM
6010	A	ACCEPTABLE PERFORMANCE
6012	A	ACCEPTABLE PERFORMANCE
6013	Ls	BIASED LOW, POSSIBLE SLOPE PROBLEM
6015	A	ACCEPTABLE PERFORMANCE
6017	WOC	WARNING:ONE RESULT ERRATIC
6018	Hs	BIASED HIGH, POSSIBLE SLOPE PROBLEM
6019	LS	BIASED LOW, PROBABLE SLOPE PROBLEM
6020	Le	BIASED LOW AND/OR ERRATIC

KEY TO SUMMARY TABLE

A	Acceptable Performance
WAI	Warning: Slight imprecision
WLI	Warning: Biased low, Probable intercept problem
WHI	Warning: Biased high, Probable intercept problem
WLS	Warning: Biased low, Probable slope problem
WHS	Warning: Biased high, Probable slope problem
WOC	Warning: Out of control - one result erratic
LI	Biased Low, Probable intercept problem
LS	Biased low, Probable slope problem
Li	Biased low, Possible intercept problem
Ls	Biased low, Possible slope problem
L	Biased low
Le	Biased low and/or erratic
HI	Biased high, Probable intercept problem
HS	Biased high, Probable slope problem
Hi	Biased high, Possible intercept problem
Hs	Biased high, Possible slope problem
H	Biased high
He	Biased high and/or erratic
OC	Out of control - one result erratic
ER	Both results erratic

FIGURE 1: LOW SPIKE (D2), REAGENT WATER

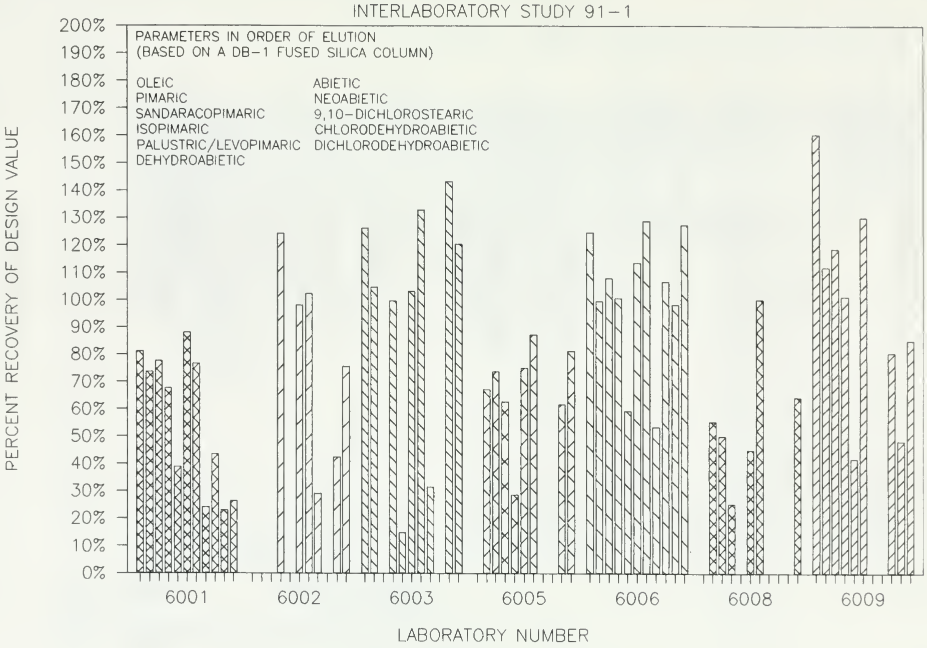


FIGURE 2: LOW SPIKE (D2), REAGENT WATER

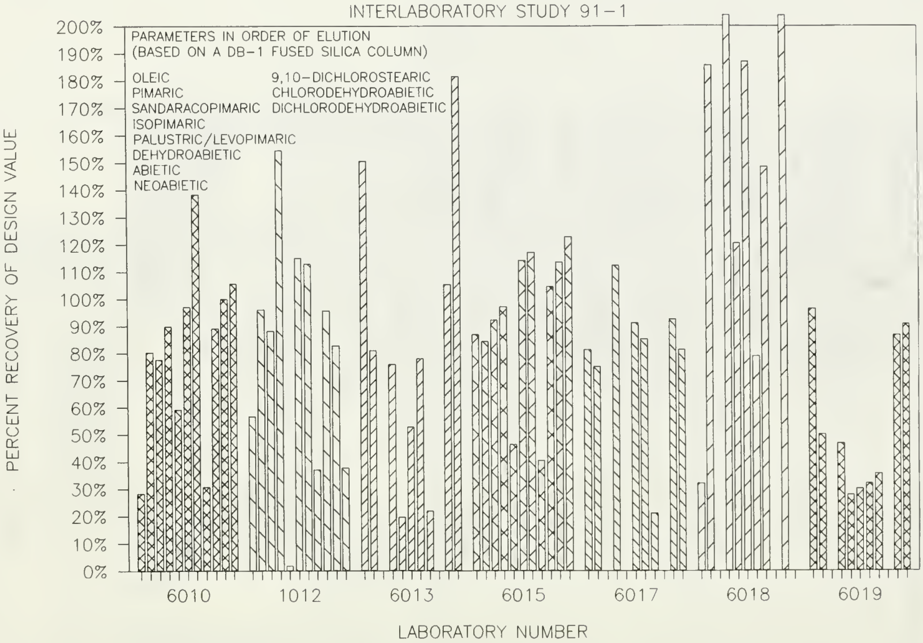


FIGURE 3: HIGH SPIKE (D3), REAGENT WATER

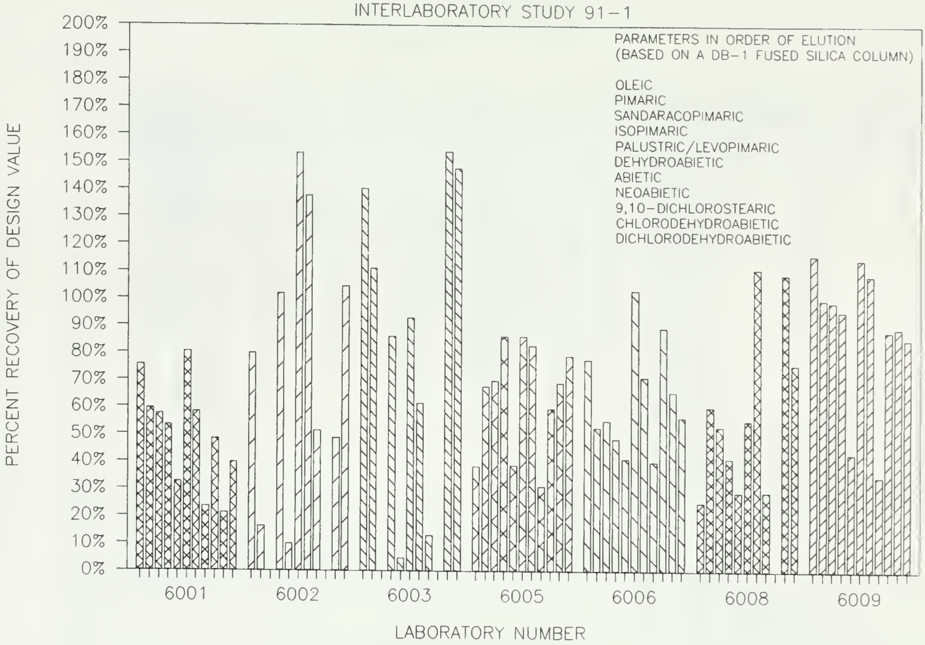


FIGURE 4: HIGH SPIKE (D3), REAGENT WATER

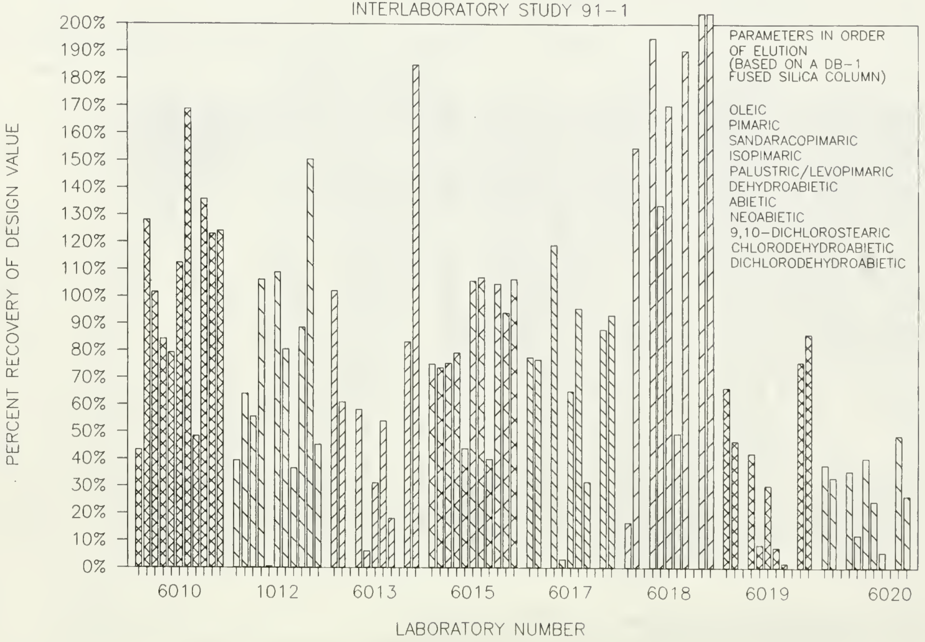


FIGURE 5: LOW SPIKE (E2), EFFLUENT

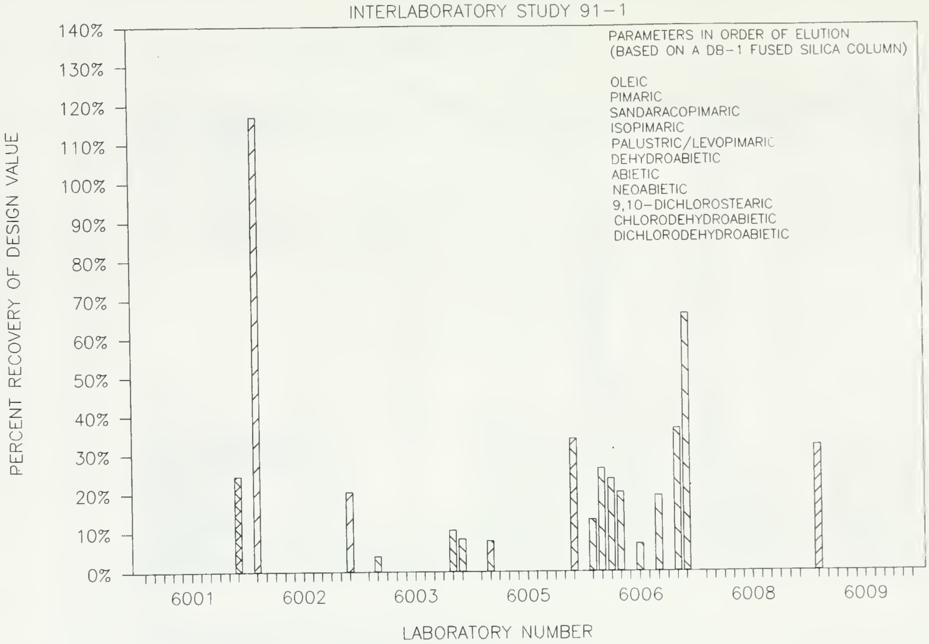


FIGURE 6: LOW SPIKE (E2), EFFLUENT

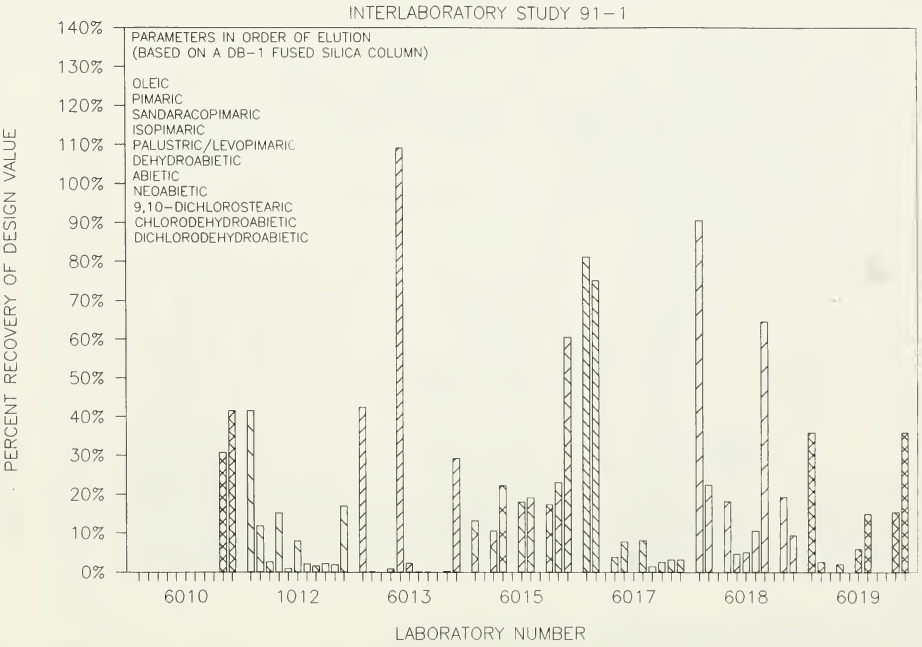


FIGURE 7: HIGH SPIKE (E3), EFFLUENT

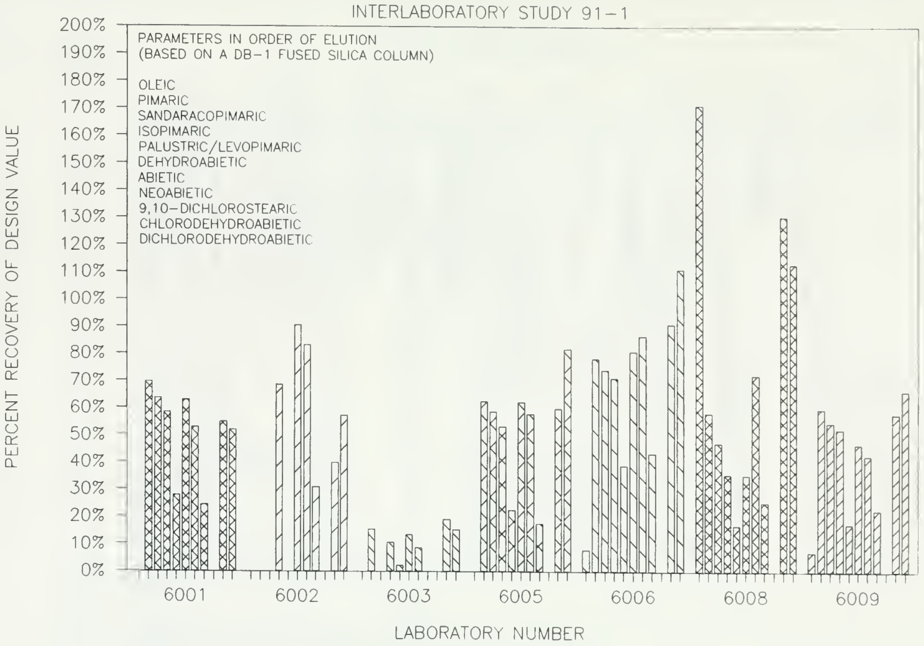


FIGURE 8: HIGH SPIKE (E3), EFFLUENT

INTERLABORATORY STUDY 91-1

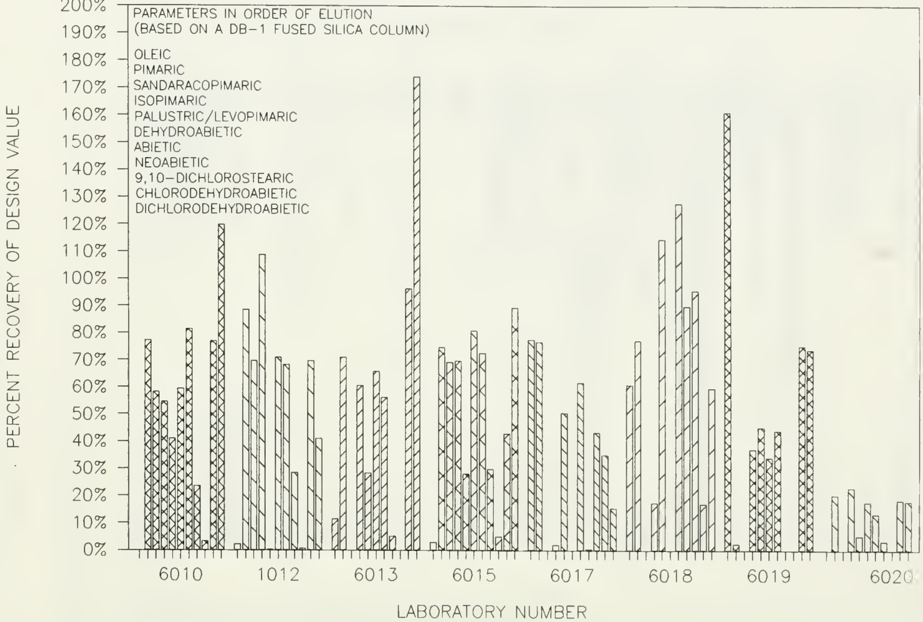


FIGURE 9: DUPLICATE SPIKE (I2), INFLUENT

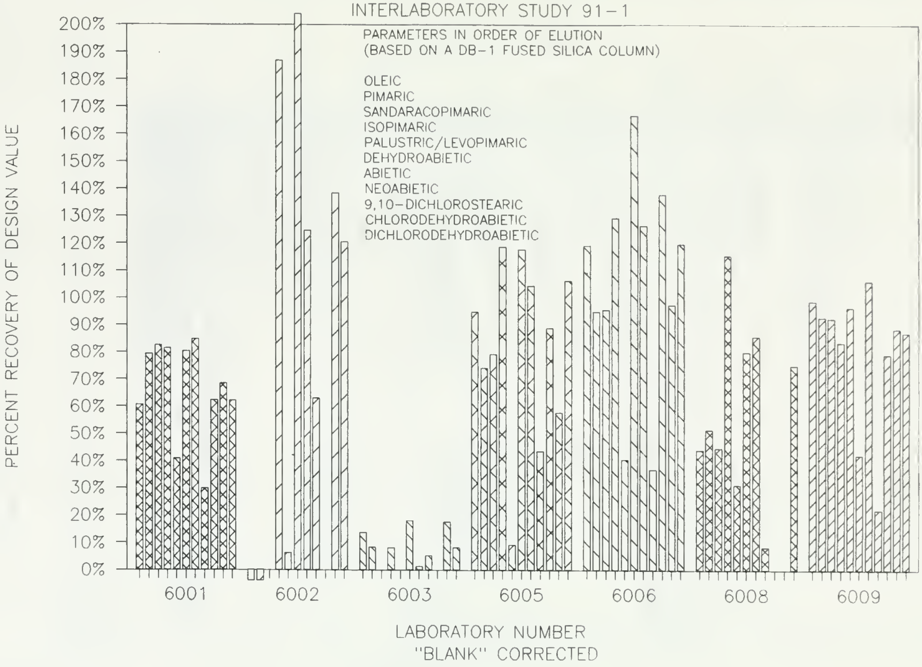


FIGURE 10: DUPLICATE SPIKE (I2), INFLUENT

INTERLABORATORY STUDY 91-1

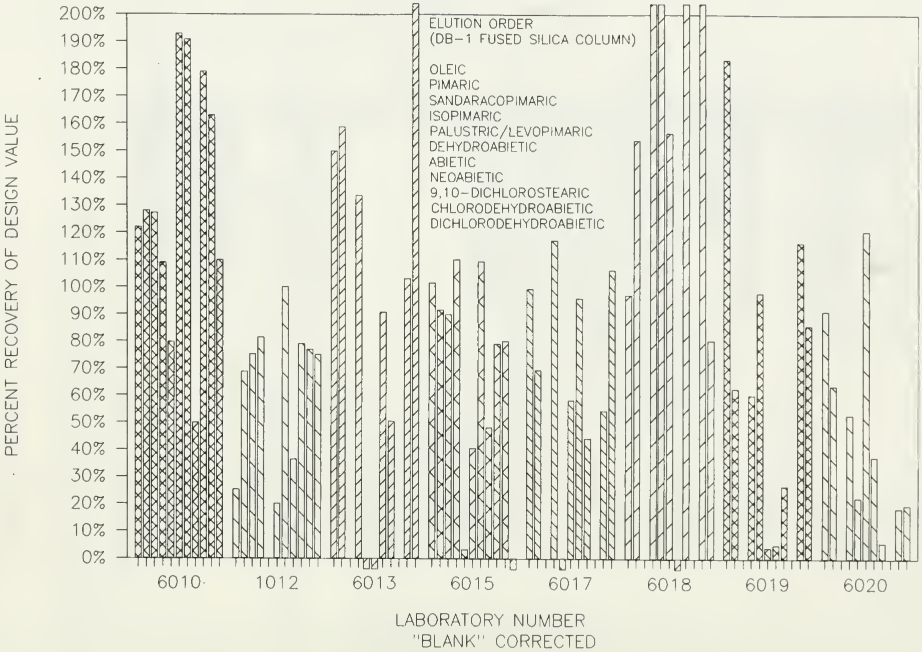


FIGURE 11: DUPLICATE SPIKE (I3), INFLUENT

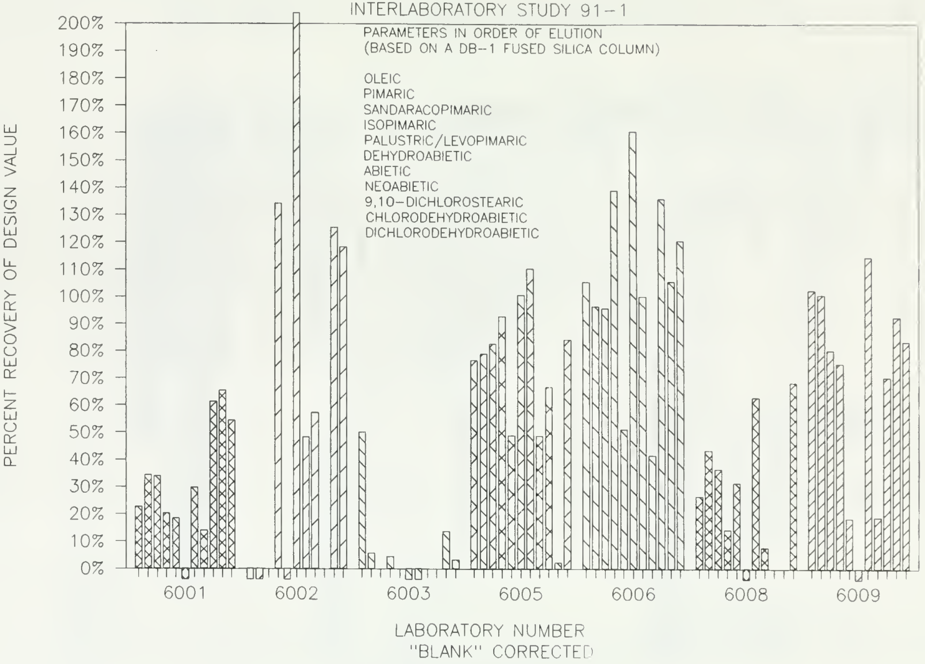


FIGURE 12: DUPLICATE SPIKE (I3), INFLUENT

INTERLABORATORY STUDY 91-1

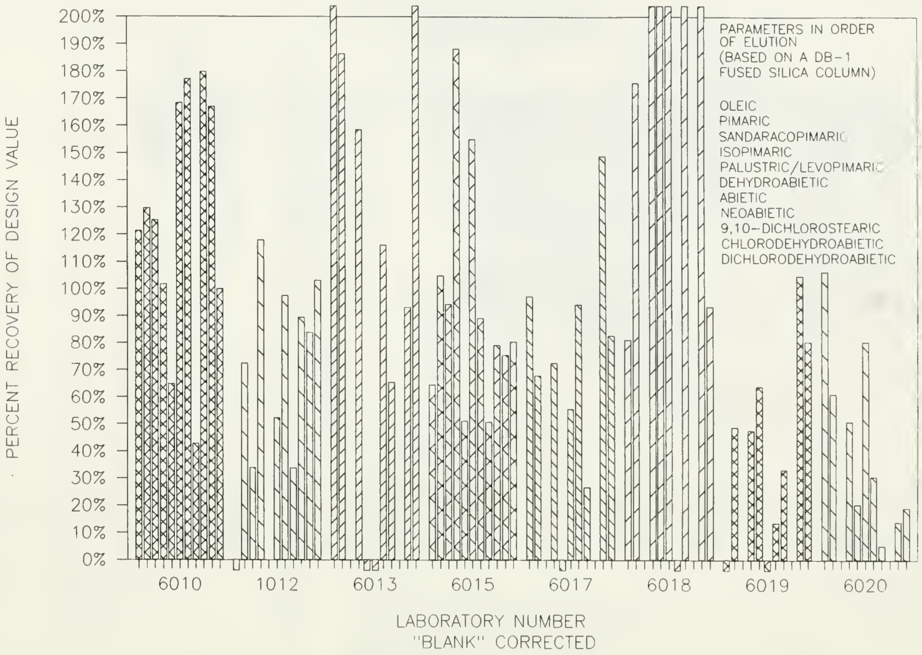
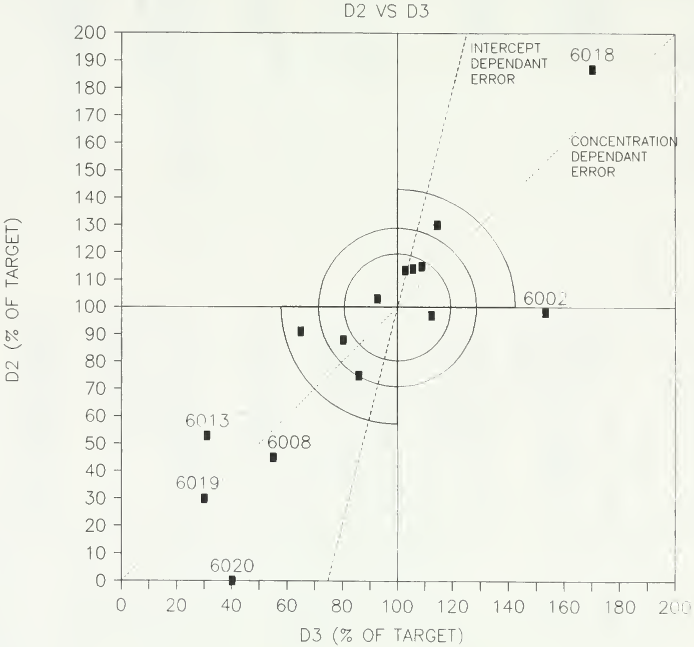


FIGURE 13: DEHYDROABIETIC ACID



7 APPENDIX 2 - LIST OF PARTICIPANTS AND CORRESPONDENCE

LIST OF PARTICIPANTS

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Contact: Ainsley Bharath
(807) 343-8505

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF RESIN AND FATTY ACIDS AND ADSORBABLE ORGANIC HALIDES

STUDY NO. 91-1

INTRODUCTION

Laboratories receiving this notification are invited by Environment Ontario, Laboratory Services Branch, to participate in an interlaboratory variability study of spiked reagent water and spiked pulp and paper mill effluent, conducted using MISA analysis protocols. Laboratories interested in participating in this program, scheduled for the week of February 18, 1991 should contact Sylvia Cussion at (416) 235-5842 of Environment Ontario to confirm participation. Written confirmation (attached response sheet) must be submitted to Environment Ontario no later than February 13, 1991 (FAX: (416) 235-5744).

BACKGROUND

This study is being conducted to assist laboratories in assessing their analytical performance. All procedures should follow those principles and protocols outlined in the MISA regulations (Ontario Reg. 695/88 as amended to Ontario Reg. 533/89). Sample sets will include three samples per matrix: a blank, one low spike (approx. 5-10 times the Regulation MDL), and a high spike (approx. 20 times the Regulation MDL).

NOTE: Any laboratory that does not have a copy of the MISA general regulations should contact Catherine Doehler at (416) 235-6055 for additional information.

The following test groups are to be included in this study:

Resin and Fatty Acids (MISA Groups 26 and PP3)

Time Limit: 7 days storage
Samples will not be preserved.

Adsorbable Organic Halides (MISA Group PP4)

Time Limit: 7 days storage
Samples will not be preserved.

SCHEDULE

During the week of February 18, 1991, participating laboratories will receive a total of nine (9) samples divided as follows: three (3) reagent water samples, three (3) each of two different effluent samples. All samples will be for the analysis of Resin and Fatty Acids. Only three samples will be designated for the analysis of Adsorbable Organic Halides.

Participating laboratories are expected to analyze the samples within the time limits specified in Schedule 2 of the general MISA regulations (Ontario Reg. 695/88). Blank reporting forms will be provided with the samples. Results for all analyses are to be reported within thirty (30) days of receipt of the samples to Sylvia Cussion at the following address:

Environment Ontario
Laboratory Services Branch
Quality Management Unit
125 Resources Rd., P.O. Box 213
Rexdale, Ontario
M9W 5L1

SUMMARY OF RESULTS

All participating laboratories will be assigned a unique identification code. All laboratories reporting results will receive a complete set of the results where they will be identified only by the identification code. Recommendations made by Environment Ontario will be included in the final report. Results will remain confidential and will only be released with the written permission of the individual participants.

It is the intent of this interlaboratory study (along with others) to assess the interlaboratory variability and detection capability for a broad range of organics and inorganics.

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF RESIN & FATTY ACIDS AND ADSORBABLE ORGANIC HALIDES

STUDY 91-1

	YES	NO
We will participate in MOE study 91-1 for the analysis of Resin & Fatty Acids and Adsorbable Organic Halides.	☐	☐

Minimum amount of sample volume required: ____________________

For the completeness of our records, and to avoid any shipping delays, please fill in the following:

Mailing Address:	Shipping Address:
____________________	____________________
____________________	____________________
____________________	____________________
____________________	____________________
____________________	____________________

Contact Person: ____

Telephone: ____

Please return this response form by February 13, 1991 to:

Sylvia Cussion
Environment Ontario
Laboratory Services Branch
Quality Management Unit
125 Resources Rd., P.O. Box 213
Rexdale, Ontario
M9W 5L1

(416) 235-5842
FAX: (416) 235-6110

Laboratory Services Branch
Quality Management Unit
125 Resources Road
Rexdale, Ontario
M9W 5L1
(416) 235-5842
FAX (416) 235-6110

February 19, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-1

Please find enclosed nine 1000 mL amber, glass bottles for the analysis of Resin and Fatty Acids and Adsorbable Organic Halides. The samples are labelled as follows:

Reagent Water Spikes:	D1	D2	D3
Treated Effluent Spikes:	E1	E2	E3
Untreated Influent Spikes:	I1	I2	I3

You should have received two of each sample. If you are missing any of the above items, please contact me at the above phone number immediately.

All samples are to be analyzed for Resin and Fatty Acids (MISA Test Groups 26 and PP3). Only the following samples are to be analyzed for Total Adsorbable Halides:

Adsorbable Organic Halide Analysis: D1 E1 I1

As stated in the notification distributed January 25, 1991, samples should be analyzed using the principles and protocols outlined in the MISA General Regulation (Ontario Reg. 695/88, as amended to Ont. Reg. 533/89). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limit for storage is 7 days.

To ensure timely release of a summary report, results are to be submitted by March 28, 1991. Report forms to be used are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact me if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

INTERLABORATORY STUDY 91-1

REPORT FORM: RESIN AND FATTY ACIDS

REPORT DUE DATE: MARCH 28, 1991

LABORATORY IDENTIFICATION CODE:

PARAMETER UNITS:	SAMPLE:	D1	D2	D3

SAMPLE PREPARATION PRINCIPLES:

INSTRUMENTAL MEASUREMENT METHOD PRINCIPLES:

GAS CHROMATOGRAPHIC COLUMN:

INTERLABORATORY STUDY 91-1

REPORT FORM: RESIN AND FATTY ACIDS

REPORT DUE DATE: MARCH 28, 1991

LABORATORY IDENTIFICATION CODE:

PARAMETER UNITS:	**SAMPLE:**	**E1**	**E2**	**E3**

SAMPLE PREPARATION PRINCIPLES:

INSTRUMENTAL MEASUREMENT METHOD PRINCIPLES:

GAS CHROMATOGRAPHIC COLUMN:

INTERLABORATORY STUDY 91-1

REPORT FORM: RESIN AND FATTY ACIDS

REPORT DUE DATE: MARCH 28, 1991

LABORATORY IDENTIFICATION CODE:

PARAMETER UNITS:	SAMPLE:	I1	I2	I3

SAMPLE PREPARATION PRINCIPLES:

INSTRUMENTAL MEASUREMENT METHOD PRINCIPLES:

GAS CHROMATOGRAPHIC COLUMN:

INTERLABORATORY STUDY 91-1

REPORT FORM: ADSORBABLE ORGANIC HALIDES

REPORT DUE DATE: MARCH 28, 1991

LABORATORY IDENTIFICATION CODE:

SAMPLE	**RESULT** **UNITS:**
D1	
E1	
I1	

SAMPLE PREPARATION PRINCIPLES:

INSTRUMENTAL MEASUREMENT METHOD PRINCIPLES:

Laboratory Services Branch
Quality Management Unit
125 Resources Road
Rexdale, Ontario
M9W 5L1
(416) 235-5842
FAX (416) 235-6110

February 19, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-1

Please find enclosed nine 1000 mL amber, glass bottles for the analysis of Resin and Fatty Acids and Adsorbable Organic Halides. The samples are labelled as follows:

Reagent Water Spikes:	D1	D2	D3
Treated Effluent Spikes:	E1	E2	E3
Untreated Influent Spikes:	I1	I2	I3

You should have received one of each sample. If you are missing any of the above items, please contact me at the above phone number immediately.

All samples are to be analyzed for Resin and Fatty Acids (MISA Test Groups 26 and PP3). Only the following samples are to be analyzed for Total Adsorbable Halides:

Adsorbable Organic Halide Analysis: D1 E1 I1

As stated in the notification distributed January 25, 1991, samples should be analyzed using the principles and protocols outlined in the MISA General Regulation (Ontario Reg. 695/88, as amended to Ont. Reg. 533/89). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limit for storage is 7 days.

To ensure timely release of a summary report, results are to be submitted by March 28, 1991. Report forms to be used are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact me if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

Laboratory Services Branch
Quality Management Unit
125 Resources Road
Rexdale, Ontario
M9W 5L1
(416) 235-5842
FAX (416) 235-5744

February 19, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-1

Please find enclosed three 1000 mL amber, glass bottles for the analysis of Adsorbable Organic Halides. The samples are labelled as follows:

Reagent Water:	D1
Treated Effluent:	E1
Untreated Influent:	I1

You should have received one of each sample. If you are missing any of the above items, please contact me at the above phone number immediately.

As stated in the notification distributed January 25, 1991, samples should be analyzed using the principles and protocols outlined in the MISA General Regulation (Ontario Reg. 695/88, as amended to Ont. Reg. 533/89). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limit for storage is 7 days.

To ensure timely release of a summary report, results are to be submitted by March 28, 1991. Report forms to be used are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact me if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

June 10, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-1

Thank you for your participation in Interlaboratory Study 91-1 for the analysis of Resin and Fatty Acids in Reagent Water and Pulp and Paper Mill Effluents and the analysis of Adsorbable Organic Halides. Please find enclosed a copy of the Tables of Results from all participants, and the target values for each parameter. Please note that there were additional compounds in the spiking mixture that are not part of MISA Group 26.

Please review the results for your laboratory for any transcription errors and report any corrections to me by no later than June 21, 1991. A final report will be prepared and forwarded to all participants by a later date.

Your Laboratory Identification Code is:

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist
(416) 235-5842